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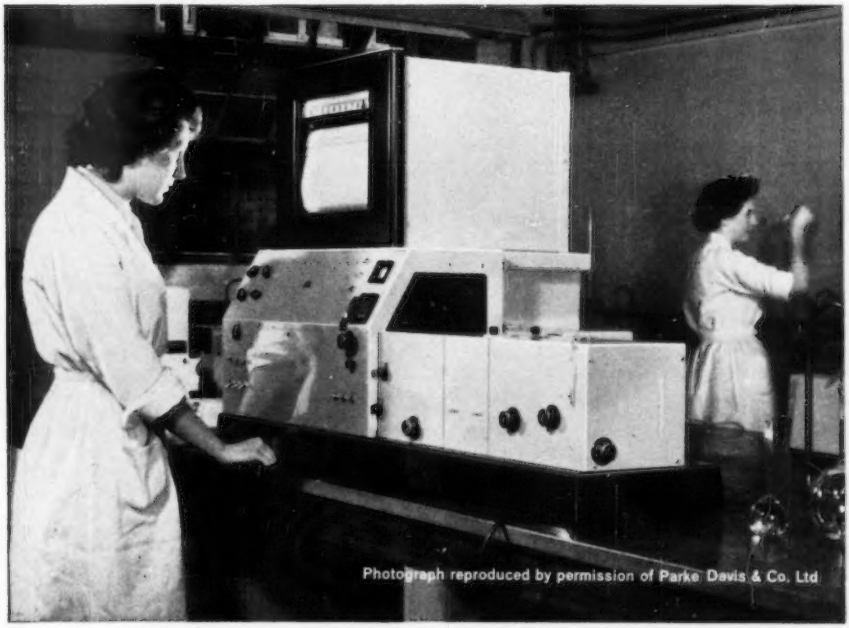

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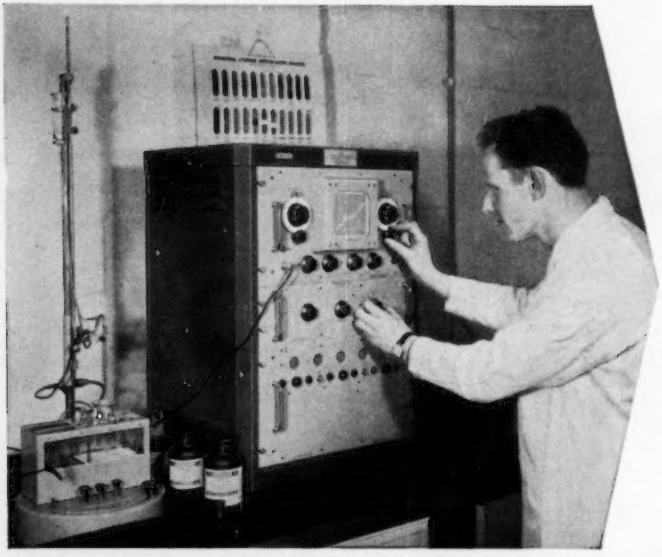

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ANALYTICAL ABSTRACTS

1.—GENERAL ANALYTICAL CHEMISTRY

General reviews of progress, reagents and methods of general application.

4918. **Proceedings of the fifteenth International Congress of Pure and Applied Chemistry (Analytical Chemistry).** Lisbon, 1956. Vol. III.—This is the last of three volumes (for Vol. I see *Anal. Abstr.*, 1959, **6**, 1175, and for Vol. II see *Anal. Abstr.*, 1960, **7**, 2031), and contains the following papers (in English unless otherwise stated).

Section VIII. Adsorption and distribution methods, pp. 9–306.—**Study of the phosphates of calcium and lead. Chromatographic and chemical analysis,** A. Badinand, R. Mallein and C. Charbonnier, 9–17 (in French). **Application of continuous electrochromatography to the separation of some characteristic anion- and cation-pairs,** C. Bighi and G. Trabanelli, 18–23 (in French). **Separation of ions of the same elements of different valencies by continuous electrochromatography,** C. Bighi and G. Trabanelli, 24–30 (in French). **Separation of co-ordination complexes by column chromatography,** J. Bjerrum, A. Jensen and F. Woldbye, 31–36. **Paper-chromatographic and paper-electrophoretic examination and separation of anionic compounds of the systems molybdenum - tungsten, niobium - tantalum and silicate - phosphate,** E. Blasius and A. Czekay, 37–49 (in German). **Partition of polyiodides between aqueous solutions and organic solvents,** R. Bock, 50–61 (in German). **Inorganic paper chromatography. Study of the variation of R_F with concentration of hydrochloric acid in n-butanol - hydrochloric acid mixtures (1 : 1),** R. A. Guedes de Carvalho, 63–86 (in French). **Electrochromatographic separation and determination of copper(II) - nickel(II) and nickel(II) - chromate ions,** L. Cavallaro, C. Bighi and G. Trabanelli, 87–91 (in French). **Separation and determination of small amounts of "acid-soluble" [organic] phosphorus compounds,** G. Ducet and I. Mencl, 92–96 (in French). **Use of paper chromatography for the characterisation of aconitine by means of its derivatives,** C. Mathis and P. Duquénois, 97–103 (in French). **Separation of polyphosphates by precipitation with various basic organic compounds and by ion exchangers,** J. P. Ebel and N. Busch, 104–113 (in French). **Experiments on retention analysis. I,** B. Erdem, 115–123 (in German). **Gradient elution theory,** E. C. Freiling, 124–138. **Determination of amino-acids by ion-exchange chromatography, using a modified Moore and Stein technique and an improved photometric method,** S. Jacobs, 140–150. **Gas chromatography with mixed fillings of chromatographic columns,** J. Janák, 151–156 (in German). **Quantitative chromato-polarographic determination of organic substances. Determination of nitro-compounds,** W. Kemula, 157–168 (in German). **Quantitative analysis with thermal-conductivity detection in gas-partition chromatography,** A. I. M. Keulemans, A. Kwantes and G. W. A. Rijnders, 169–185. **Coulometry applied to gas-phase chromatography,** A. Liberti, 195–207. **Identification of amino-acids by paper chromatography,** A. Lima, 208–210 (in Portuguese). **Technique for limiting the destruction of amino-acids of cereals in chromatographic studies,** A. Lima, 211–212 (in Portuguese). **Influence of pH on the paper-electrophoretic separation of alkaloids,** G. B. Marini-Bettòlo and J. A. Coch Frugoni, 213–220 (in Italian). **Use of paper chromatography for the analysis of food dyes,** I. Netto, 221–229 (in French). **Chromatographic analysis of the products of oxidation of porphyrin,** R. A. Nicolaus, L. Mangoni and L. Caglioti, 230–239 (in Italian). **Detection and identification of phenols by paper chromatography. Water as chromatographic solvent,** E. A. H. Roberts, 240–243. **[Paper-chromatographic] study of cynarin in plants in the presence of other caffeyl derivatives,** M. L. Scarpati and G. Oriente, 246–251 (in Italian). **Determination of the *N*-terminal amino-acid in a peptide chain as the 3,4-dihydro-7-nitroquinoxalin-2-one,** E. Scoffone, E. Vianello and A. Lorenzini, 252–255 (in French). **Application of partition chromatography to the transuranic elements, with particular reference to plutonium,** M. R. English and J. K. Foreman, 256–271. **Apparatus for vapour-phase chromatography: analysis of pyridine homologues: separation of the isomers of picoline,** W. J. Murray and A. F. Williams, 272–287. **New chromatographic methods for routine application in the explosives industry,** J. W. Erskine and A. F. Williams, 288–305.

Section IX. General, pp. 309–697.—**Determination of traces of impurities in gases with sodium biphenyl,** A. Antonini, 309–315 (in French). **Quantitative mineralogical analyses of mixtures of iron oxides, free or combined, by selective oxidation - reduction,** J. Aubry and P. Vayssière, 316–319 (in French). **Quantitative analysis of calcium aluminate,** J. Barlot, 320–322 (in French). **Separation of three unknown reducing steroids from the urine of pregnant women,** E.-E. Baulieu and M. de Vigan, 323–326. **Physico-chemical studies on pure polyoxyethylene derivatives. Possibilities of analytical applications,** J. Bolle, P. Ragon and T. Jullig, 327–337 (in French). **A cause of error in the determination of nitrogen in special steels by the wet procedure,** R. Castro, J. Allemand and R. Poussardin, 346–351 (in French). **Crystallochemical analysis and the Barker index of crystals,** L. W. Codd, 352–362. **Critical study of the determination of vitamin C in foods,** B. V. J. Cuvelier, M. E. Hansens and J. Bovy, 385–394 (in French). **New rapid gravimetric procedure for the determination of iron,** J. Dick, 403–408 (in German). **Precision and accuracy,** A. H. Dodd, 409–416. **Determination of gaseous elements in metals and refractory materials,** R. M. Fowler, 428–442. **Co-precipitation of radium with barium sulphate,**

L. Gordon and K. Rowley, 455–465. **The value of correlation calculations in the interpretation of analytical results,** Y. Graff, 466–479 (in French). **Determination of oxygen-containing functional groups in blown asphaltic bitumens and similar substances,** J. Knotnerus, 480–493. **Determination of potentials not directly measurable,** J. Lefebvre, 494–500 (in French). **Volumetric oxidations with chlorine dioxide and its behaviour as a pseudo-halogen,** G. R. Levi, 540–546 (in Italian). **Apparatus for the rapid combustion of volatile organic liquids for the determination of impurities containing sulphur or halogens,** F. Martin and A. Floret, 547–563 (in French). **New methods and techniques for the determination of binary or ternary mixtures of sulphurous and sulphuric anhydrides and hydrogen sulphide in a gaseous medium,** G. Pannetier and C. Meltzheim, 565–577 (in French). **Graphical evaluation of indirect analyses in impure binary mixtures,** W. Posch, 578–583 (in German). **Application of pyrohydrolysis to the determination of fluorine in thorium, uranium and plutonium fluorides,** P. Regnaut and J. Corpel, 590–595 (in French). **Sodium hexametaphosphate as a reagent in quantitative analysis,** R. Ripan *et al.*, 596–601 (in French). **New colour indicators for use in very basic media,** R. Schaal, 610–614 (in French). **Graphical calculation for the evaluation of indirect analyses,** E. Schwarz-Bergkampf, 619–622 (in German). **Notes on classical adsorption indicators and new analytical applications,** F. Sierra Jiménez, 630–655 (in Spanish). **Adsorption indicators of the phenosafranine type,** F. Sierra Jiménez, 656–664 (in Spanish). **Melting curves (temperature heat content curves) as criteria for purity,** W. M. Smit, 665–678. **New form of iodimetric determination of sulphite or of alkaline mixtures of sulphite, sulphate and sulphide,** H. N. Terem and A. Isnel, 680–684 (in French). **Pimento paste. Detection of added tomato paste,** E. S. L. Tavares de Castro, 696–697 (in Portuguese).

Section X. Standardisation of methods and various applications, pp. 701–937.—**Quality control in the iron and steel industry and standardisation of methods of analysis,** C. J. Arlidge, 701–708. **Determination of keten and its derivatives,** P. Besson and G. Rémond, 709–718 (in French). **Determination of ammonia and ammonium salts in certain substances capable of decomposition with evolution of ammonia in the presence of fixed alkalis,** G. Bourjol, 728–739 (in French). **Colorimetric determination of chlorates in hypochlorites with Lunge's reagent,** G. Bourjol and J. Sourd, 740–745 (in French). **Automatic determination of nitric oxide in coke-oven gas,** H. W. Deinum and W. J. Hanssen, 746–765. **Sodium carbonate as a volumetric standard,** W. C. Easterbrook, 773–787. **Spectrographic analysis of iron ore for ordinary and special elements by the solution spark technique,** T. Ikegami and T. Takei, 789–808. **Determination of zinc in metallurgical products,** J. Kinnunen and B. Wennerstrand, 809–818. **Oscillographic method of periodical polarisation for alkali- and hydrogen-ion analysis,** P. Kirkov, 819–830. **Identification and determination of the new laxative drug di-(*p*-acetoxyphenyl)-2-pyridylmethane [bisacodyl],** A. M. Leal and M. A. Constantino, 831–832 (in Portuguese). **New methods of determination of chlorpromazine in pharmaceutical preparations,** M. Lopes and A. M. Leal, 833–837 (in Portuguese). **New analytical determination of dysproteinaemia,** J. Marquevielle and F. Tayeau, 838–847 (in French). **New method of acetylation: phosphoric acetylation,** P. Mesnard and M. Bertucat, 848–862 (in French). **Determination of resins in fatty substances,** A. J. Morais, 863–870 (in Portuguese). **Determination of acetic anhydride in mixtures with acetic acid,** J. J. Nicholls and T. Ellerington, 871–878. **Characterisation of aerosols by thermal precipitation,** C. F. Schadt and R. D. Cadle, 899–906. **Development and operation of a vacuum-fusion gas-analysis apparatus for use in the quality control of ferrous materials,** K. Speight and G. M. Gill, 907–918. **The coulomb as a basis for analysis,** P. S. Tutundžic, 919–924 (in German). **Detection, characterisation and determination of aldehydes. II. Derivatives formed by reaction with 1,2-di-(*p*-methoxybenzylamino)ethane and 1,2-dianilinoethane,** S. Veibel and I. G. K. Andersen, 925–932 (in French). **Determination of nitrofurazone and furoxone in veterinary feeding-stuffs,** G. Zwingelstein and J. Jouanneteau, 933–937 (in French).

4919. **Variations and errors in experimental investigations.** S. Lewin (South-West Essex Coll. of Technol., London, E.17). *Lab. Practice.*—**I. Some general causes,** 1961, **10** (2), 99–101. **II. Compromise errors,** 1961, **10** (3), 162–164. **III. Insufficient formulations and inaccurate assumptions,** 1961, **10** (6), 363–366, 408. **IV. Limitations of chemical tools and techniques,** 1961, **10** (7), 474–477. **V. Personal and characteristic errors,** 1961, **10** (8), 556–558.

4920. **Increasing the selectivity of analytical reactions by masking.** K. L. Cheng (Metals Div., Kelsey-Hayes Co., New Hartford, N.Y.). *Anal. Chem.*, 1961, **33** (6), 783–790.—A review with 20 references is presented. G. S. ROBERTS

4921. **Use of polarography for continuous and automatic control of laboratory and manufacturing processes.** Ya. I. Tur'yan and A. M. Murenkov. *Zavod. Lab.*, 1961, **27** (5), 507–517.—A review with 51 references is presented. G. S. SMITH

4922. **Potentiometric and polarometric [amperometric] titrations using tetraphenylborates. II. Mercurimetric determinations.** A. Heyrovský (II Med. Clinic, Charles' Univ., Prague, Czechoslovakia). *Coll. Czech. Chem. Commun.*, 1961, **26** (5), 1305–1312 (in German).—Both potentiometric and amperometric titration can be used for determining tetraphenylborate with Hg^{II}. Each mole of Na tetraphenylborate consumes 4 moles (8 equiv.) of $Hg(NO_3)_2$ giving 4 moles of phenylmercury nitrate, 3 moles of HNO_3, and 1 mole each of H_2BO_3 and $NaNO_3$. The potentiometric titration is reproducible in spite of the formation of intermediates (phenylboronic acid and diphenylmercury); the first break in the curve occurs after consumption of 3 equiv. of Hg^{2+} and the second after 8. The polarogram shows a double wave after the addition of 3 equiv. of Hg, owing to reduction of phenylmercury chloride; the wave then increases linearly until 8 equiv. of Hg have been added, when no further increase occurs. Both $Hg(NO_3)_2$ and $Hg(ClO_4)_2$ are suitable and are used in 0·1N-Na acetate buffer soln.; $HgCl_2$ forms insol. intermediates, and is less satisfactory. Titrations in the presence of Br^-, I^- and SCN^- are also affected by precipitation, and the presence of acetone is undesirable. The method is suitable for the determination of cations (1 to 5 mg), *e.g.*, K, Th and Ag, choline and penta- or deca-methonium, with an accuracy within ±1%. End-point determinations are less easy than in the argentimetric method. J. P. STERN

See also Abstracts—**4924**, Nitrosochromotropic acid as a reagent. **4941**, Use of 8,8′-biquinolyl disulphide. **4942**, Use of Daxim as reagent. **4981**, 1-Hydroxycyclohexanecarboxylic acid in pptn. of Zr. **5003**, Chlorphosphonazo III as reagent. **5013**, *OO*-Diethylphosphorodithioic acid as reagent for Re. **5321**, Sensitivity of spectrographic analysis.

2.—INORGANIC ANALYSIS

General, determination of elements (arranged in the order of the Periodic Table), analysis of minerals and inorganic industrial products.

4923. **Separation and micro-identification of metal ions by the ring-oven technique. I.** Eric John Singh and Arun K. Dey (Chem. Lab., Univ. of Allahabad, India). *Mikrochim. Acta*, 1961, (3), 366–369 (in English).—The separation of Pb^{II}, Hg^{I}, Ag^{I}, Cu^{II}, Cd^{II}, Hg^{II}, Bi^{III}, As^{III}, Sb^{III}, Sn^{II} and Co^{II}, by the ring-oven technique, with oxalate as complexing agent, has been studied. With 60% ethanol as solvent the following separations were made—Hg^{I} - Ag - Pb; Cu - Cd - Hg^{II}; Cu - Cd - Pb; Cu - Cd - Bi; Hg^{II} - Pb - Cu - Cd; Cu - Cd - Hg^{II} - Pb - Co; As - Sb - Sn^{II}. The best concn. of oxalate vary according to the metals to be separated. R. M. ROWLEY

4924. **Tests for metal ions and fluoride with mononitrosochromotropic acid.** Sachindra Kumar Datta and Sachindra Nath Saha (Victoria Coll., Coochbehar, W. Bengal, India). *Mikrochim. Acta*, 1961, (3), 361–365 (in English).—Nitrosochromotropic acid gives stable rose to violet-coloured compounds with many metals, including Cu^{II}, Pd^{II}, Co^{II}, Ni^{II}, Ce^{III}, La^{III}, Y^{III}, Th^{IV} and Zr^{IV}. A sensitive, but not specific, spot test for Cu, Co, Ni and Pd is described. Place 1 drop of 0·02% reagent soln. (pH 3·5) on a spot-plate, and add 1 drop of the metal soln. and 1 drop of aq. NH_3 soln. A violet colour is observed with 1×10^{-6} g of Cu, 5×10^{-6} g of Pd, 4×10^{-6} g of Ni, or 3×10^{-6} g of Co at a final pH of ≃8. To test for F^-, place 1 drop of 0·01% reagent soln. on a spot-plate, and add 1 drop of soln. containing 0·8 to 1·7 μg of Th, Ce, La or Y, and 1 drop of 0·01% hexamine soln. Then add 1 drop of the test soln. The colour changes from pink or violet to yellow or orange with 1 μg of F^- at a pH between 3·8 and 5·2. R. M. ROWLEY

4925. **Separation of radio-elements by chromatography on paper impregnated with an inorganic ion exchanger.** J. P. Adloff (Centre de Rech. Nucl., Dépt. de Chim. Nucl., Strasbourg-Cronenbourg, France). *J. Chromatography*, 1961, **5** (4), 366–367 (in French).—Paper was impregnated with zirconium phosphate (Alberti *et al.*, *Anal. Abstr.*, 1961, **8**, 1293), or with hydrated zirconium oxide or zirconium tungstate by similar methods. The paper was washed and dried and cut into strips (12 cm × 2 cm). Development was by the descending method in an atmosphere satd. with water vapour at 60°. Solvents were either H_2O, HCl (0·1N or 0·5N), NH_4NO_3 (0·05N or 0·1N) or NH_4Cl (0·1N or 1N). The running time varied between 5 and 30 min. Detection was by a Geiger - Müller counter. A table of R_F values and separations achieved is given. A good separation was effected between $^{204}Tl^{+}$ and $^{204}Tl^{3+}$ and between ^{223}Ra and ^{223}Fr. G. RUSSELL

4926. **Paper-chromatographic methods of separation of copper, nickel and cobalt from one another and their detection.** M. R. Verma and P. K. Gupta (Nat. Phys. Lab., Hillside Road, New Delhi). *Curr. Sci.*, 1961, **30** (1), 10–11.—Spots (≃0·005 ml) of soln. (0·02 to 0·002M) of sulphates of the metals named were loaded on to Whatman No. 1 paper and dried. They were then treated with 40% aq. KSCN soln. (≃0·01 ml), the paper was dried, rolled into a cylinder, placed in the trough containing the solvent, covered with a bell-jar and developed for 2 to 3 hr. The paper was then dried and the metals were detected with acetone (for Co; the blue spot fades rapidly and must be marked with a pencil), 1% ethanolic dimethylglyoxime soln. (for Ni; permanent red spot) and 1% ethanolic dithio-oxamide soln. (for Cu; permanent grey spot). Compact spots were given with butanol - pyridine - aq. NH_3 (8:2:1) or n-pentanol - pyridine - aq. NH_3 (8:2:1) as solvent. The positions of the spots in ascending order are Cu, Co, Ni; the Co and Ni spots are close together, and may overlap. To obtain compact spots the amount of KSCN must be <100 times that required to complex all the metal. R. E. E.

4927. **Determination of ultra-micro amounts of lead, copper and zinc in bases and acids by means of amalgam polarography on a stationary mercury drop.** S. I. Sinyakova and I. V. Markova (V. I. Vernadskii Inst. of Geochem. and Anal. Chem., Acad. Sci., USSR). *Zavod. Lab.*, 1961, **27** (5), 521–525.—Electrolysis is carried out with a sessile mercury drop (1·3 to 1·6 mm) on an upturned capillary tube. For Cu and Pb the potential is −1·0 V, and for Zn −1·6 V, in a soln. 1·75M in NaOH and 0·1M in KCl. The stripping is carried out by reducing the potential at the rate of 0·4 V per min. G. S. SMITH

4928. **Analytical scheme for the individual separation of zirconium, cerium, neodymium and traces of thorium from solutions containing aluminium.** C. G. Wallace (U.K.A.E.A. Research Group, C.37 Royal Arsenal, Woolwich, London). A.E.R.E. Report, AERE-R3575, 1961. 10 pp.—The development of the procedure given in AERE-AM 73 (*Anal. Abstr.*, 1961, **8**, 2771) is described. The experiments involved the use of radioactive measurements to determine the behaviour of Th in the procedure, but weighing was used in the study of Zr, Ce and Nd. The recoveries of Th, Zr, Ce and Nd were 96, 93, 96 and 97%, respectively. G. J. HUNTER

4929. **Colorimetric determination of phosphorus, arsenic, germanium and silicon by use of molybdate. III. Optimum conditions for the development of colour in the molybdenum blue method.** Teru Yuasa. *Rep. Gov. Chem. Ind. Res. Inst., Tokyo*, 1961, **56** (2), 43–50.—A detailed study is reported of variations of the method, the colour being developed under various conditions by means of ammonium molybdate and hydrazine sulphate, and the blank soln. being treated in the same way. It is shown that the intensity of the colour is greatly influenced by the concn. of MoO_4^{2-} and pH of the sample soln., and that better results are obtained when the following relationship is satisfied—

$$\log [Mo] = -a.pH + b,$$

where [Mo] is the concn. of MoO_4^{2-} and a and b are constants. Values for a and b are, for P, 1·79,

1·79; for As, 1·15, 1·15; and for Ge, 1·21, 1·48; values for Si are not given. The effects of such variables as hydrazine sulphate concn. and temp. of development on the intensity of the colour are discussed. I. E. JONES

4930. **Direct spectrophotometric determination of niobium, titanium and tungsten with quinol using a background correction technique.** J. P. McKaveney (Crucible Steel Co. of America, Pittsburgh, 13, Pa.). *Anal. Chem.*, 1961, **33** (6), 744–747.—*Procedure*—A suitable sample is treated with 50 ml of HCl (1:1), 25 ml of solvent mixture [85% H_3PO_4 - 95% H_2SO_4 - H_2O (10:3:12)] and 5 ml of HNO_3. The soln. is evaporated to fumes and heated for a further 1 min. If dark, the soln. is oxidised dropwise with HNO_3 and again evaporated to fumes. When cool, the soln. is diluted with 25 ml of H_2SO_4 (35%), and transferred to a 50-ml flask where it is treated with 10 ml of $SnCl_2$ soln. (88 g of $SnCl_2.2H_2O$ dissolved in 60 ml of HCl plus 50 g of tartaric acid in 100 ml of H_2O diluted to 250 ml) and diluted to 50 ml. After 15 to 60 min., a 1-ml aliquot is added to 25 ml of quinol reagent (15 g dissolved in 500 ml of 95% H_2SO_4, stored in amber glassware). After a further 10 ±2 min., the extinctions are measured at 400 and 500 mμ; the extinctions at these wavelengths of a background soln. (a 1-ml aliquot of sample soln. in 25 ml of 95% H_2SO_4) and of 95% H_2SO_4 are also measured. Each of the elements Nb, Ti and W can be determined satisfactorily in alloys, either alone or when present as the combinations Nb and W or Nb and Ti. Care must be taken in the preparation of the quinol reagent; nitrate must be removed. J. H. WATON

4931. **Chromium, cobalt and strontium in some [U.S. National] Bureau of Standards rock reference samples.** K. K. Turekian and M. H. Carr (Dept. of Geology, Yale Univ., New Haven, Conn., U.S.A.). *Geochim. et Cosmoch. Acta*, 1961, **24** (1–2), 1–9.—A method for the simultaneous determination of Cr and Co in rocks by neutron activation is described in detail, Cr being pptd. as $BaCrO_4$ and Co as cobaltinitrite. Counting is done on a single-channel γ-ray spectrometer using the Co 1·33 MeV and Cr 0·32 MeV γ-peaks. The possible errors involved are evaluated. Strontium is determined by emission spectrography, using Sr 4067 A, calibrated by standards analysed by isotope dilution. R. A. HOWIE

4932. **Determination of chromate, arsenate, periodate, iodate, bromate and chlorate ions in the presence of one another.** L. Szekeres (Univ. of Agric. Sci., Budapest). *Ann. Chim., Roma*, 1961, **51** (2), 200–206.—Six aliquot portions of a soln. of the mixture are treated with various reagents and in all cases the iodine liberated from KI is finally titrated with standard $Na_2S_2O_3$ soln. The concn. of the six oxidising agents are obtained from suitable equations. The principles of the separation depend on the following characteristics—I^- are not oxidised by AsO_3^- or ClO_3^- in N-H_2SO_4; IO^- are reduced to I^-, and IO_4^- to IO_3^-, by alkaline H_3O_3; BrO_3^- and ClO_3^- with HBr give Br, which can be reduced by H_2O_2 or urea (in the presence of IO_3^-) to BrO^-; Cr^{3+} are oxidised to CrO_4^{2-} by alkaline H_2O_2. L. A. O'NEILL

4933. **Coulometric titration of iron, antimony and tin with electrolytically generated permanganate ion.** A. I. Kostromin and S. A. Flegontov (V. I. Ul'yanov-Lenin Kazansk State Univ.). *Zavod. Lab.*, 1961, **27** (5), 528–530.—The titration of small amounts (0·05 to 5 μg) of Fe, Sb and Sn in 100 ml of soln. (7N-H_2SO_4 containing 2·5 ml of N-$MnSO_4$) is described. G. S. SMITH

4934. **Determination of water in [inorganic] compounds by the hydride method.** G. M. Toptygina (N. S. Kurnakov Inst. Gen. and Inorg. Chem., Acad. Sci., USSR, Moscow). *Zhur. Anal. Khim.*, 1961, **16** (2), 201–204.—The method described by Elitsur (*cf. Zavod. Lab.*, 1946, **12**, 794; abstr. in *Analyst*, 1948, **73**, 298) is shown to give high results in a number of cases, since the hydroxides formed in the reaction are themselves capable of reacting with CaH_2 with liberation of H. J. W. PRICE

4935. **Inorganic thin-layer chromatography. III. Separation of the alkali group.** H. Seiler and W. Rothweiler (Inst. f. Inorg. Chem., Univ. Basel). *Helv. Chim. Acta*, 1961, **44** (4), 941–942 (in German).—The thin-layer method was applied to the chromatographic separation and identification of the cations in the alkali group, with violuric acid as reagent for identification. The cations tested were Li^+, Na^+, K^+ and Mg^{2+}. M. SULZBACHER

4936. **Spectral determination of the isotopic composition of lithium.** A. N. Zaĭdel', A. N. Razumovskiĭ and M. P. Chaika. *Optics and Spectroscopy*, 1961, **10** (1), 7–9; English translation of *Optika i Spektroskopiya*, 1961, **10** (1), 15–18.—In order to determine the proportion of ^6Li in a sample (≃0·05 mg of LiCl), the components of the 6707·8-A line are measured with a photo-electric spectrometer incorporating a Fabry - Perot interferometer. The hollow-cathode discharge tube used to excite the spectrum is described. Calibration is effected with standard samples. The sensitivity is 0·05% of ^6Li and the coeff. of variation is 0·15 to 0·7% for ^6Li contents between 40 and 90%. The analysis takes 10 to 15 min. B. S. COOPER

4937. **Rapid determination of potassium in common salt and other saline products by precipitation and titration of its tetraphenylborate.** B. Riva (Univ. Cagliari, Italy). *Ann. Chim., Roma*, 1961, **51** (1), 52–63.—The K is pptd. from acidified aq. soln. with aq. Na tetraphenylborate at 0° and the ppt. is filtered off, washed and dissolved in acetone. The acetone soln. is then treated with satd. aq. $HgCl_2$ soln., in the presence of a known excess of alkali. A soln. of KI and a known excess of acid are then added and the acid is back-titrated with standard alkali to methyl red. Other ions likely to be present in salt do not interfere. L. A. O'NEILL

4938. **Flame-photometric methods of determining the potassium in potassium tetraphenylborate.** M. G. Reed and A. D. Scott (Agron. Dept., State Univ., Ames, Iowa, U.S.A.). *Anal. Chem.*, 1961, **33** (6), 773–775.—Three rapid methods are described. (*i*) The ppt. of K tetraphenylborate (**I**) in aq. soln. can be dissolved by adding acetone and the soln. examined in a flame photometer. (*ii*) The ppt. of **I** is heated for 20 min. at 350° and is thereby rendered sol. in water. (*iii*) The ppt. of **I** is decomposed by boiling with an aq. soln. of $HgCl_2$ to produce a soln. of KCl, which can be subjected to flame photometry. G. S. ROBERTS

4939. **Separation of milligram amounts of caesium from a large excess of ammonium salts.** A. G. Pratchett (U.K.A.E.A. Research Group, C.37,

Royal Arsenal, Woolwich, London, S.E.18). A.E.R.E. Report AERE-R3404, 1961. 8 pp.—The development of the procedure is described. In an experiment involving a column of Zeo-Karb 315 (−16 +50 mesh, 100 cm long, 0·79 sq. cm cross-section), 1200 ml of soln. containing 320 g of NH_4Cl, 20 mg of Cs and 250 g of NaOH giving a pH of 13 was used. At a flow rate of about 3 ml per min. satisfactory retention of the Cs was achieved; any Na^+ and NH_4^+ that were adsorbed were removed with 9 column-volumes of 0·25N-HNO_3 before the elution of the Cs with 4 column-volumes of 3N-HCl; the loss of Cs was about 4%. The presence of 10 g of K resulted in a 15% loss of Cs. The separation takes 10 hr.

G. J. HUNTER

4940. **Analytical applications of chelating agents. XVIII. Spectrophotometric and absorptiometric micro-determination of copper with 1,2-di-(2-aminoethoxy)ethane-*NNN'N'*-tetra-acetic acid.** F. Bermejo Martínez and M. Paz Castro (Fac. de Cienc., Univ. de Santiago de Compostela, Spain). *Rev. Soc. Quim. Mex.*, 1961, **5**, 58–60.—*Cf. Inf. Quim. Anal.*, 1959, **13** (4), 94; *Anal. Abstr.*, 1960, **7**, 1667. E. C. APLING

4941. **Analytical application of 8-mercaptoquinoline (thio-oxine) and its derivatives. XVII. 8,8'-Biquinolyl disulphide, a new selective reagent for the photometric determination of small amounts of copper.** E. Bankovskiĭ, A. F. Ievin'sh, E. O. Luksha and P. Ya. Bochkans (Inst. Chem., Acad. Sci. LatvSSR, Riga). *Zhur. Anal. Khim.*, 1961, **16** (2), 150–157.—The reagent (**I**) forms a stable complex with Cu^I over a wide pH range in the presence of an excess of hydroxylamine (pH 4 to 11·5) or ascorbic acid (pH 2·19 to 13). Soln. of the complex in $CHCl_3$ show an absorption max. at 432 mμ, and Beer's law is obeyed for Cu concn. up to 8 μg per ml. Most of the common metals may be present in large excess, but Au must be absent. High concn. of the common anions do not interfere, but SCN^- and CN^- must be absent. *Procedure*—Adjust the sample soln. to pH 1 to 2 and add 50% Na acetate soln. to bring the pH to 5 to 6. Add 50% hydroxyammonium chloride soln. (5 ml) or 20% ascorbic acid soln., and extract the Cu by shaking vigorously for 3 to 4 min. with 5 or 10 ml of a 0·2% $CHCl_3$ soln. of **I**. Separate the $CHCl_3$ layer and measure its extinction in a 1-cm cell. For the determination of traces of Cu in the presence of Fe, Al, Sb, Bi, Ti, Zr, Cr and Ge, add 20% Na tartrate soln. (3 to 4 ml) and extract at pH 4 to 5. Nickel and Co in the presence of Fe will be co-extracted if shaking is prolonged beyond 3 min. *Separation of Cu from dilute soln.*—To 500 ml of soln. add 10% ascorbic acid soln. (10 ml) and 20% Na acetate soln. (20 ml) and a soln. of **I** in pyridine (25 mg per ml). Filter, dry the ppt. at 120°, re-dissolve it in $CHCl_3$ and measure the extinction. (*Cf. Anal. Abstr.*, 1960, **7**, 3628).

J. W. PRICE

4942. **New selective micro-methods for the analysis of metals. I. Analytical use of a new organic reagent 4-imino-1,3-dimethylalloxan 5-oxime (Daxim). Copper determination.** K. Burger (L. Eötvös Sci. Univ., Budapest). *Magyar Kém. Foly.*, 1961, **67** (5), 208–212.—The use of 4-imino-1,3-dimethylalloxan 5-oxime (**I**) (prep. described) is suggested for the determination of metals. Its acidic dissociation constant has been established by potentiometric titration (pK = 8) and the composition and stability of its copper complex have been studied. An absorptiometric method for the determination of micro amounts (1 to 13 μg per ml) of Cu is described. *Procedure*—The test soln. is evaporated to 1 to 2 ml and neutralised with 2N-HCl or 2N-NaOH. A 1% soln. of **I** in formaldehyde (0·4 ml) and aq. NH_3 - NH_4Cl buffer soln. (pH 8) (5 to 6 ml) is then added. After removal of any ppt. by centrifuging, the pH of the soln. is adjusted to 7 to 9 and the soln. is made up to 10 ml with water. The extinction of the soln. is measured at 382 mμ. The extinction is directly proportional to the Cu content of the soln. No interference is caused by Hg^{2+}, Pb, Bi, Mn, Zn, Fe^{3+} or Al, but Co, Pd, Ni and Fe^{2+} must be absent. The error is within ±3%. B. VASZKO

4943. **Iodimetric amperometric determination of copper(II).** D. Singh and V. S. Agarwala (Electrochem. Lab., Hindu Univ., Benares). *Curr. Sci.*, 1961, **30** (1), 12–13.—Bivalent copper oxidises iodides with pptn. of CuI according to the equation $2Cu^{2+} + 5I^- = 2CuI + I_3^-$, and the resulting soln. is titrated with standard $Na_2S_2O_3$ soln. at pH 3·2 to 5·2 at constant voltage with a pair of polarised electrodes. Up to 6·3 mg of Cu^{2+} can be determined with an error within ±1·5%. Silver, Pb, Bi, Sn^{IV}, Al, Zn and Ni do not interfere; Fe^{3+} can be masked with alkali pyrophosphate.

R. E. E.

4944. **Paper-chromatographic method for the quantitative determination of copper and zinc 8-hydroxyquinolinates.** T. D. Miles, A. C. Delasanta and J. C. Barry (Textile, Clothing and Footwear Div., Quartermaster Res. & Engng Command, Res. & Engng Center, Natick, Mass., U.S.A.). *Anal. Chem.*, 1961, **33** (6), 685–687.—After extraction (*e.g.*, from treated textile materials) with 10% H_2SO_4 and then with $CHCl_3$ at pH 5 to 7, Cu 8-hydroxyquinolinate is chromatographed with butanol - HCl - H_2O (8:1:1) on circular Whatman No. 3 paper. Two rings, one due to 8-hydroxyquinoline and Cu^{2+} (R_F = 0·7) and one to Cu^{2+} alone (R_F = 0·4), are formed. Both rings are cut out, and the Cu is extracted with dilute acetic acid and titrated with EDTA, with PAN [1-(2-pyridylazo)-2-naphthol] as indicator. A $CHCl_3$ soln. of Zn 8-hydroxyquinolinate is chromatographed as for the Cu complex. When dry, the area between the solvent front and the 8-hydroxyquinoline ring (R_F = 0·7) which contains the Zn ring (R_F = 0·8) is cut out, extracted with H_2O and titrated in ethanol - H_2O (1:1) soln. with EDTA, with murexide as indicator. Good agreement is shown with results by other methods.

J. H. WATON

4945. **Polarographic determination of silver with a platinum electrode.** I. A. Kedrinskiĭ (Lensovet Leningrad Tech. Inst.). *Zavod. Lab.*, 1961, **27** (5), 538–540.—A cell of small vol. (1 ml) with a sintered-glass base forms the top part of a calomel electrode. A rotating platinum wire placed in the cell is used to determine Ag polarographically in a soln. 0·1N in aq. NH_3 and 0·1N in NH_4Cl. G. S. SMITH

4946. **Determination of impurities in beryllium by activation analysis using remote reactor facilities.** A. P. Seyfang and R. Todd (U.K.A.E.A. Production Group, Springfields, Lancs.). U.K.A.E.A. Report PG172(S), 1961. 26 pp.—Aspects considered include the apparatus required and interferences

resulting from reactions other than simple (n, γ) ones. Of the elements that may be determined, 58 are tabulated together with information on radiations emitted and limits of detection. Also listed are those elements for which the method is not applicable. G. J. HUNTER

4947. **Quantitative determination of traces of barium, nickel, copper, antimony, molybdenum, manganese, cadmium, tin, gold and arsenic in beryllium metal by the radioactivation method.** V. R. Negina and V. N. Zamyatnina. *Zhur. Anal. Khim.*, 1961, **16** (2), 209–212.—The method is based on the irradiation of the sample, containing the impurities in the concn. range of 10^{-3} to 10^{-5} %, together with a standard sample, for 3 hr. in a flux of $0{\cdot}5 \times 10^{13}$ neutrons per sq. cm per sec., followed by chemical separations and measurement of the activities of the separated elements. *Determination of As, Au, Sn^{IV}, Mn^{II} and Cd*—Dissolve the sample by heating under reflux in 10N to 12N-HCl, add 20 to 30 mg of each impurity as carrier, and extract the diethyldithiocarbamate (**I**) complexes of As, Au and Sn^{IV} into $CHCl_3$ from the strongly acid soln. Reduce the acidity to N and extract the **I** complexes of Mn^{II} and Cd into $CHCl_3$. Evaporate the first extract to dryness, decompose the residue with HNO_3 and again evaporate to dryness. Re-dissolve the residue in HCl and precipitate Au and As as sulphides from 6N to 8N-HCl and Sn as sulphide from N-HCl. Transfer the Mn and Cd from the second extract into HNO_3 (1:3) and precipitate CdS and MnO_2. Re-dissolve all the ppt. and finally isolate for measurement as $Mg_2As_2O_7$, Au, SnS_2 and $Mn_2P_2O_7$. *Determination of Ba, Ni, Cu, Sb and Mo*—Dissolve the irradiated sample as described above, adding 20 to 30 mg of each element as carrier. Dilute to an acidity of 3N, precipitate $BaSO_4$, reduce the Sb to Sb^{III} with hydrazine and extract the **I** complexes into $CHCl_3$. Evaporate the extract to dryness, treat it with HNO_3 and again evaporate. Dissolve the residue in 4% aq. NH_3 (Sb is insoluble), add dimethylglyoxime and extract the Ni complex from this soln. into $CHCl_3$ and the Cu complex from the neutralised soln. Precipitate MoS_3 from weakly acid soln. Re-dissolve all the ppt. and finally isolate as $BaCrO_4$, NiO, CuO, Sb_2O_4 and MoO_3. Recoveries of 60 to 80% are obtained.

J. W. PRICE

4948. **A polymethine dyestuff as a specific reagent for magnesium in spot tests.** V. Anger (Forschungslab. der Lobachemie, Wien, Austria). *Mikrochim. Acta*, 1961, (3), 512–515 (in German).—The condensation product of glutaconaldehyde and barbituric acid is red in alkaline soln., and fluoresces strongly. With Mg^{2+} it forms a blue salt or lake, which does not fluoresce. It does not form salts with other ions in alkaline soln. When it is used for spot tests the limit of detection is 0·1 μg of Mg in 1 drop of soln. The preparation of the reagent is described. R. M. ROWLEY

4949. **Extraction of magnesium with 8-hydroxyquinoline.** S. J. Jankowski and H. Freiser (Dept. of Chem., Univ., Pittsburgh, Pa., U.S.A.). *Anal. Chem.*, 1961, **33** (6), 776–781.—The extraction of Mg with 8-hydroxyquinoline into organic solvents such as isopentyl alcohol, ethanolamine and $CHCl_3$ - 2-butoxyethanol is reported. The effect of quaternary ammonium salts on the extraction of the Mg complex is studied and an outline of a method for the determination of μg amounts of Mg in the presence of Ca, Ba and Sr is given.

G. S. ROBERTS

4950. **Micro-analysis of aqueous solutions of salts. I. The use of critical miscibility temperature for determining the concentrations of solutions of zinc salts.** F. Gölles (Inst. f. Pharmakog., Univ. Graz, Austria). *Mikrochim. Acta*, 1961, (3), 487–497 (in German).—The suitability of a number of liquids for the determination of zinc sulphate, acetate, nitrate and chloride has been studied, and related to the solubility of the salts. Both sensitivity and reproducibility depend on the form of the P *vs.* c curve, where $P = 100\alpha/(1 + \alpha)$, and α and c are the partition coefficient and concentration of the salt. R. M. ROWLEY

4951. **Polarographic determination of cadmium in an acid sulphate - iodide supporting electrolyte.** V. A. Oshman and A. P. Chistyakova (Ural Sci. Res. and Development Inst. of the Copper Industry). *Zavod. Lab.*, 1961, **27** (5), 532–536.—For the determination of Cd in ores and concentrates, the sample [0·1 to 2 g according to the concn. (5 to 0·0005%)] is heated with HCl followed by a mixture of HNO_3 and HCl (3:1) or of HNO_3 and Br, or, for slags, with HNO_3, HF and H_2SO_4. The soln. is evaporated to dryness, and As, if present, is removed by heating with 8 to 20 ml of HCl, 0·1 to 0·3 g of KBr and 0·2 to 1 g of hydrazine sulphate. The dry residue is treated with 3 to 15 ml of H_2SO_4 (1:1) and, if As has been removed, with 1 to 5 ml of HNO_3. The soln. is evaporated to fuming, water is added, and the soln. is evaporated nearly to dryness. The cooled residue is treated with 2 ml of H_2SO_4 (1:1), 1 to 3 g of KI and 20 to 40 ml of water and boiled until the vol. has been reduced to 20 ml. In this process salts of Sn are hydrolysed and pptd. Small amounts (totalling 0·1 to 0·2 g) of NaH_2PO_2 are added to decolorise the hot soln., but prolonged heating, whereby H_2SO_4 would be reduced to H_2S, is avoided. The soln. is washed into a flask with water containing 2 ml of H_2SO_4 (1:1) and cooled. After addition of 0·5 ml of 0·5% gelatin soln. and dilution to 25 ml the soln. at 50° to 60° is treated with reduced iron and filtered within 20 to 30 min. into the polarographic cell. The height of the wave at −0·63 V *vs.* the S.C.E. or −0·35 V *vs.* the mercury pool is measured. No interference is caused by Pb, Sn, Sb, Bi, Te, Cu, In, Mo or W.

G. S. SMITH

4952. **Determination of mercury by means of radioactive indicators.** T. Năsçuţiu (Inst. de Fiz. Atomică, Bucureşti, Romania). *Acad. R.P.R., Stud. Cercet. Chim.*, 1960, **8** (4), 649–652.—The Hg^{2+} in soln. are converted into $[HgI_4]^{2-}$ by addition of excess of KI soln. labelled with ^{131}I. The compound $[CuX][HgI_4]$, where X is ethylene- or propylene-diamine, is pptd. by addition of $[CuX_2]NO_3$ soln. and its amount is deduced from the radioactivity of the ppt. As little as 0·08 mg of Hg can be determined with an accuracy within ±5%. By using $Fe(OH)_3$ to entrain the ppt., even smaller amounts of Hg may be determined, but the accuracy falls to ±10%.

D. R. DUNCAN

4953. **Determination of traces of mercury in rocks and soils in the presence of other cations. I. Spectrophotometric monochromatic method.** F. Popea and M. Jemăneanu (Lab. de Geochim. al

Întreprind. de Prospecț. și Lab., București, Romania). *Acad. R.P.R., Stud. Cercet. Chim.*, 1960, **8** (4), 607–616.—The sample (1 to 5 g) is dissolved by being heated under reflux until colourless with 20 to 30 ml of conc. H_2SO_4 and a wt. of HNO_3 equal to that of the sample. After cautious dilution of the soln. with 70 to 75 ml of H_2O, nitrous fumes, if present, are destroyed by adding $KMnO_4$ and the soln. is diluted to 250 ml. A suitable aliquot is transferred to a 300 to 500-ml separating-funnel and the pH is adjusted to between 4 and 5 with aq. NH_3 followed by an acetic acid - Na acetate buffer. EDTA (disodium salt) (25 ml of an 8% soln., to mask heavy metals) and 5 ml of satd. KSCN soln. (to mask Ag) are added. A 0·0006% soln. of dithizone in CCl_4 is added from a micro-burette, with shaking, until the orange colour due to Hg dithizonate changes to green. The CCl_4 layer is washed first with ammoniacal EDTA, then with EDTA soln. containing acetic acid, and filtered, and the extinction at 500 mμ is measured. This method may be used for the determination of 2 to 20 μg of Hg.

II. Direct colorimetric spot method. F. Popea and M. Jemăneanu. *Ibid.*, 1960, **8** (4), 617–622.—The method is similar to that described in Part I, except that the sample is dissolved by boiling with conc. HCl and bromine water, and the amount of Hg is determined from the colour of the CCl_4 layer after adding specified amounts of the soln. of dithizone in CCl_4. The accuracy is sufficient for the geochemical prospecting of mercury ores. The minimum detectable quantity is 0·1 μg of Hg, and the limit of dilution 1 in $2·5 \times 10^6$.

D. R. DUNCAN

4954. **Polarographic determination of boron. Further studies of the mannitol - sulphite system. Application of the cathode-ray polarograph.** G. F. Reynolds and E. A. Terry (Chem. Inspectorate, Royal Arsenal, Woolwich, London, S.E.18, England). *J. Polarographic Soc.*, 1961, **7** (1), 2–9.—A polarographically-reducible compound is liberated by the action of mannitol - boric acid on Na_2SO_3 which can be used to determine B (*cf.* Lewis, *Analyst*, 1956, **81**, 531). The wave-height (first wave) was proportional to the square of the B concn. On a pen-recording polarograph, the useful range was up to 300 μg per ml; 2 μg per ml could be detected. An increase in sensitivity was obtained by using a cathode-ray polarograph, when 2 μg per ml could be determined and, by reducing the Na_2SO_3 concn. from 0·12M to 3mM, 1 μg of B per ml could be determined. The sensitivity was further increased to 0·1 μg per ml by the addition of HCl to pH 3·9. G. RUSSELL

4955. **Direct fluorimetric determination of aluminium with haematoxylin.** P. P. Solodovnikov (Kazan Aviation Inst.). *Zhur. Anal. Khim.*, 1961, **16** (2), 237–240.—*Procedure*—To a soln. containing 2 to 10 mg of Al add 40% acetic acid (1 ml) and dilute to 25 ml with water. Heat to 50° to 60°, saturate the soln. with NaCl, then add 1 to 2 g of Na acetate and titrate at 50° to 60° with 0·2N-KF, with haematoxylin (1% in ethanol) as indicator, to a colour change from violet to yellow. To avoid pptn. of basic salts add small amounts of Na acetate during the titration, so that the pH at the end-point is between 4·5 and 5·2. The titration is reversible. A 50-fold excess of Mg and a 10-fold excess of Ca, Mn and Zn can be tolerated, but large amounts of Cu, Fe, tartrate, citrate and oxalate interfere. J. W. PRICE

4956. **Polarographic determination of lead in high-purity aluminium.** E. N. Vinogradova, L. N. Vasil'eva and K. Iobst (M. V. Lomonosov Moscow State Univ.). *Zavod. Lab.*, 1961, **27** (5), 525–527.—The sample (0·2 to 2 g) is dissolved in HCl in the presence of mercury and the soln. is evaporated nearly to dryness. Water is added and evaporated off several times to remove the excess of acid, and the soln. of the residue is acidified to a pH of 5 and diluted to 100 ml. Electro-deposition is carried out from 50 ml of soln. on to a stationary mercury drop placed on a gold contact; interference by Cd is thus prevented. After electrolysis for 60 min. at −0·8 V and an interval of 30 to 45 sec., the anodic stripping polarogram from −0·8 to 0 V is obtained. A standard soln. of Pb is added and the process is repeated with a fresh mercury drop.

G. S. SMITH

4957. **Determination of sodium in aluminium alloys with the sodium-glass electrode.** R. S. Chernyak (Moscow Aviation Technol. Inst.). *Zavod. Lab.*, 1961, **27** (5), 536–537.—The glass of the electrode contains 25% of Na_2O, 9% of B_2O_3, 5% of Al_2O_3 and 61% of SiO_2. The bulb contains 0·1N-NaCl and a silver chloride electrode. The potential in an unknown soln. is compared with that in a standard soln. of Na^+. The difference corresponds to the ratio of the concn. of Na^+. The sample of aluminium (1 to 5 g) is transformed into oxide by treatment with small amounts, totalling 1 to 2 ml, of 0·3 to 0·5% $HgCl_2$ soln. The residue is treated with water and subjected three times to electrodialysis (Tsŷvina, *Trudŷ Komiss. Anal. Khim.*, 1952, **4**, 274) and the soln. is diluted to 50 ml. The potential of the special glass electrode in this soln. is determined *vs.* a calomel electrode.

G. S. SMITH

4958. **Fluorimetric determination of gallium in zinc.** G. I. Kuchmistaya. *Zavod. Lab.*, 1961, **27** (4), 377–379.—The sample (1 g) is dissolved in 20 ml of HCl (1 : 1) in the presence of 0·5 ml of 2% NaCl soln. (to avoid loss of Ga). The cooled soln. is treated with 0·1 to 0·15 ml of 5% $TiCl_3$ soln. [5 g of titanium dissolved in 100 ml of HCl (1 : 1)] and then shaken with 20 to 25 ml of butyl acetate. The extract is washed with HCl (1 : 1) (2 × 3 to 4 ml) and then extracted twice with 10 to 15 ml of water. The aq. soln. is mixed with 0·5 ml of 2% NaCl soln. and evaporated to dryness. The residue is dissolved in 3 to 5 ml of HCl (1 : 1) and treated with 0·1 to 0·15 ml of the 5% $TiCl_3$ soln., 2 ml of benzene, 0·5 ml of butyl acetate, and 0·6 ml of 0·5% rhodamine B soln. [250 mg in 50 ml of HCl (1 : 1)]. The mixture is shaken for 2 min. The fluorescence intensity of the filtered extract is compared with a scale of standards (0 to 0·06 μg of Ga). The method is sensitive to 10^{-4} % of Ga in zinc. G. S. SMITH

4959. **Polarometric [amperometric] determination of indium.** J. Doležal and J. Zýka (Inst. for Anal. Chem., Charles' Univ., Prague). *Coll. Czech. Chem. Commun.*, 1961, **26** (5), 1464–1466 (in German).—The In is determined as $In(ClO_4)_3$ at pH 4·6 by amperometric titration with EDTA (disodium salt); the limit of sensitivity is 5×10^{-5}M-In^{III} and the accuracy is high. Titration with $K_4Fe(CN)_6$ or Na diethyldithiocarbamate is less useful and titration with Na_3PO_4 or 8-hydroxyquinoline is useless. J. P. STERN

4960. **Volumetric determination of thallium with tetraphenylborate. I.** F. Bermejo Martínez and R. Martínez de la Riva (Univ., Santiago de Compostela, Spain). *Inf. Quim. Anal.*, 1961, **15** (2), 31–35.—The solution containing Tl^{3+} is treated with an excess of a standard solution of Na tetraphenylborate and the excess measured by titration with a standard solution of a quaternary ammonium salt, with Titan yellow (C.I. Direct Yellow 9) as indicator. L. A. O'NEILL

4961. **Polarographic determination of small amounts of thallium in cadmium.** E. K. Prokof'ev. *Zavod. Lab.*, 1961, **27** (5), 530–532.—For the determination of Tl (<0·003%) in cadmium oxide the sample (2·5 g) is dissolved in 5 ml of HCl (4:1) and the soln. is boiled for 5 to 10 sec. with 1 or 2 drops of 10% $K_4Fe(CN)_6$ soln., followed by treatment with 13 ml of 25% aq. NH_3, in 0·5 to 1·0-ml portions, with vigorous shaking to dissolve the pptd. $Cd(OH)_2$. The cooled soln. is then treated with 2·5 ml of 0·1N-NaOH or, if Pb is present, with 5 ml of 1·5M-K Na tartrate together with 1·5 ml of 5N-NaOH, and diluted to 25 ml. After removal of O with 0·3 g of Na_2SO_3 the polarogram for Tl is obtained. For metallic cadmium, 2·0 g is dissolved in 15 ml of HCl (1:3) in the presence of H_2O_2, added dropwise. The soln. is evaporated until it begins to crystallise, 10 ml of water is added and the evaporation is repeated. This operation is repeated twice more, the residue is dissolved in 5 ml of water, the soln. is boiled with 1 or 2 drops of 10% $K_4Fe(CN)_6$ soln., 12 ml of 25% aq. NH_3 is added in portions, and the procedure is continued as described above. G. S. SMITH

4962. **Determination of impurities in high-purity thallium by radioactivation.** I. P. Alimarin, Yu. V. Yakovlev, M. N. Shchulepnikov, D. A. Vlasov, G. M. Chernov and Yu. A. Surkov (V. I. Vernadsky Inst. Geochem. and Anal. Chem., Acad. Sci. USSR, Moscow). *Zhur. Anal. Khim.*, 1961, **16** (2), 213–216.—Manganese, Cu, Zn, As and Sb, in the concn. range of 10^{-5} to 10^{-7} %, are determined without chemical separation after irradiation for 20 to 48 hr. in a flux of $0·87 \times 10^{13}$ neutrons per sq. cm per sec. by using a 50-channel γ-scintillation spectrometer and comparing with standards. Standards are prepared by irradiating micro amounts of a salt of each impurity, adding 5 to 10 mg as carrier, and, after chemical separation, isolating as $Mn_2P_2O_7$, CuSCN, $ZnHg(SCN)_4$, As and Sb_2O_4. J. W. PRICE

4963. **Determination of yttrium-90 and lanthanide fission products in reactor fuel processing and effluent treatment plant solutions.** U.K.A.E.A. (Prod. Group, Tech. Branch, Windscale, Cumb.). U.K.A.E.A. Report PG206(W), 1961. 7 pp.—Full experimental details are given. The rare-earth-metal fluorides are co-pptd. with LaF_3. After pptn. of $Ce(IO_3)_4$ and $Zr(IO_3)_4$ the La is pptd., first as hydroxide and then as oxalate, which is β-counted through an effective absorber of 5 mg per sq. cm; the counter is standardised with a source of ^{90}Sr - ^{90}Y. The chemical yield of La is determined by titration of the La oxalate with $KMnO_4$ soln. G. J. HUNTER

4964. **Radiochemical separation of the lanthanides in fission products.** F. Clanet (Commissariat à l'Énergie Atomique, D.A.M. Essais, Bruyères-le-Châtel, S. et O., France). *J. Chromatography*, 1961, **5** (4), 356–362 (in French).—Place in a centrifuge tube (100 ml), as carriers, 10 mg of each of the cations sought. Add the soln. (in HNO_3) of the fission products followed by 50 mg of Zr carrier and 0·15M-$KBrO_3$ (5 ml). Heat on a water bath for 30 min., then add conc. HF (5 ml), continuing the heating for 5 min. Centrifuge and wash the ppt. twice with 0·1M-HF (10 ml). Dissolve the ppt. in the minimum vol. of HNO_3 and satd. H_3BO_3 soln. Dilute to 20 to 30 ml, add 5 mg of Ba as hold-back carrier, then NH_4Cl and aq. NH_3 to pH 9. Centrifuge and dissolve the ppt. in the minimum of conc. HCl. Pass this soln. through a column (6 mm × 30 cm) of Dowex 2 (Cl^- form) (200 to 400 mesh). Wash the column with at least one column-vol. of conc. HCl. Evaporate the eluate to small vol. and repeat the hydroxide pptn. and column treatment. Finally dissolve the ppt. in the minimum vol. of 0·1N-HCl. This soln. is separated on a column (7 mm × 60 cm) of Dowex 50 (NH_4^+ form) (200 to 400 mesh). The column is operated at 95° and gradient elution is carried out with ammonium lactate soln. at pH 5; 3-ml fractions are collected. The actual gradient depends on the separation desired, for which values are given. The lanthanides in the fractions are pptd. as oxalates, which are ignited to oxides and these are transferred to cellulose membrane discs (10 mm diameter) for counting. An over-all yield of 80 to 85% is claimed. G. RUSSELL

4965. **Determination of radio-cerium.** G. J. Hunter and M. Perkins (U.K.A.E.A. Research Group, C.37, Royal Arsenal, Woolwich, London, S.E.18). A.E.R.E. Analytical Method, AERE-AM69, 1961. 16 pp.—Full details are given of a procedure that relates particularly to the determination of ^{141}Ce and ^{144}Ce in soln. of fission products. The chemical separation is based on the one involving successive pptn. of $Ce(IO_3)_4$, $Ce(OH)_3$ and cerous oxalate, which is ignited to CeO_2 to produce sources that are β-counted through an aluminium absorber of ≃200 mg per sq. cm and γ-counted through an absorber of ≃1500 mg per sq. cm. The activities of ^{141}Ce and ^{144}Ce are calculated from the two count rates. G. J. HUNTER

4966. **Analysis of borides of the transition elements and rare-earth elements.** L. N. Kugai and T. N. Nazarchuk (Inst. Metallo-Ceramics and Special Alloys, Acad. Sci. USSR, Kiev). *Zhur. Anal. Khim.*, 1961, **16** (2), 205–208.—The determination of free B in borides of Ti, Zr, Cr, W and the rare-earth metals by selective dissolution in soln. of $Ce(SO_4)_2$ or KIO_3 was found to be impossible owing to the partial solubility of the borides, but free B can be determined in zirconium boride by dissolution in a mixture of H_2O_2 and HNO_3. *Procedure*—Dissolve the sample (1 g) in 10% H_2O_2 soln. (20 ml) and HNO_3 (10 drops) by heating under reflux for 30 min. Filter, and neutralise the filtrate to methyl red with NaOH soln. Titrate the boric acid in the presence of mannitol with 0·1N- or 0·01N-NaOH to phenolphthalein. *Analysis of rare-earth-metal hexaborides*—Dissolve the sample (0·5 g) by heating under reflux with HNO_3 (1:1) (20 ml). Dilute to 250 ml and determine the rare-earth metal in a suitable aliquot by titration with EDTA (disodium salt), with arsenazo as indicator. To another aliquot add sufficient EDTA to complex the rare-earth metal, neutralise with NaOH soln. and determine the boric acid as described above. J. W. PRICE

4967. New technique for the separation of tervalent actinide elements from lanthanide elements. F. L. Moore (Oak Ridge Nat. Lab., Tenn., U.S.A.). *Anal. Chem.*, 1961, **33** (6), 748–751.—The actinide elements (Am and Cm) are preferentially extracted in 2 min. as MCl_5^{2-} from HCl soln. (0·1M) containing LiCl (≃12M) by a 20% soln. of tri-iso-octylamine in xylene. The order of decreasing extractability is Am and Cm, Th, Eu and Pm, Ce, and Y and Tm. Applications of this procedure to the separation of Am from Pm and of Am and Cm tracers from lanthanide tracers are discussed. The procedure should be suitable for rapid analytical separation and general purification work.

J. H. WATON

4968. Determination of potassium thiocyanate with sodium hypobromite in alkaline medium. A. Claeys and H. Sion (Lab. de Chim. Anal., Univ. Ghent, Belgium). *Bull. Soc. Chim. Belg.*, 1961, **70** (5–6), 197–203.—Low concn. of KSCN can be determined by adding a mixture of the sample soln. (25 ml) and 2N-NaOH (2 ml) to an excess (50 ml) of standard soln. of BrO^- and, after 1 hr., titrating the excess of BrO^- iodimetrically in H_2SO_4 soln. with aq. $Na_2S_2O_3$. The concn. of SCN^-, BrO^- and $Na_2S_2O_3$ should be ≃0·01 to 0·1M. The precision is within 0·15%. A standard soln. of BrO^-, stable for at least one month, can be prepared by the addition of NaOH to a soln. of $KBrO_3$ and KBr in dil. H_2SO_4.

W. J. BAKER

4969. Determination of ammonium thiocyanate with sodium hypobromite in alkaline medium. A. Claeys and H. Sion (Lab. de Chim. Anal., Univ. Ghent, Belgium). *Bull. Soc. Chim. Belg.*, 1961, **70** (5–6), 213–217.—The procedure previously described for the determination of KSCN (*Anal. Abstr.*, 1961, **8**, 4968) can be applied to the determination of NH_4SCN (0·007 to 0·1M). The error is within ≃0·1%.

W. J. BAKER

4970. Spectrographic analysis of silicon tetrachloride for trace amounts of boron. T. J. Veleker and E. J. Mehalchick (Chem. and Metall. Div., Sylvania Electric Prod., Inc., Towanda, Pa., U.S.A.). *Anal. Chem.*, 1961, **33** (6), 767–770.—The determination of 0·8 to 50 parts per 10^9 of B in $SiCl_4$ is achieved by pre-concentration followed by emission spectrographic analysis. The concentration stage consists in partial hydrolysis of the sample with aq. methyl cyanide soln.; the silica and co-pptd. boric acid are then packed into graphite electrodes. The spectra are produced by a d.c. arc with a Stallwood jet and argon gas. The coeff. of variation is 14%.

G. S. ROBERTS

4971. Co-precipitation of germanium with hydroxides of iron and other metals. Kh. Ya. Kuus (Tartu State Univ.). *Zhur. Anal. Khim.*, 1961, **16** (2), 166–170.—Germanium in concn. of 2 to 10 μg per ml can be quant. recovered from $Fe(OH)_3$ ppt. when the Fe to Ge ratio is ≮25:1. The Ge can be extracted from the ppt. by washing with 10% Na_2S soln. Magnesium hydroxide and manganese hydroxide can also be used as co-precipitants. The completeness of recovery was followed radiometrically by means of soln. containing ^{71}Ge.

J. W. PRICE

4972. Methods for the sampling and analysis of tin and tin alloys. Part 10. Iron (photometric method). British Standards Institution (2 Park Street, London, W.1). B.S. 3338: Part 10:1961. 7 pp.—The use of 1,10-phenanthroline is specified.

4973. Determination of ultra-micro amounts of zinc, lead and copper in high-purity tin by amalgam polarography. A. G. Stromberg, M. S. Zakharov, V. E. Gorodovykh and L. F. Zaichko (Tomsk Polytech. Inst.). *Zavod. Lab.*, 1961, **27** (5), 517–521.—A stationary mercury drop is prepared on a platinum wire, 0·2 mm in diameter and 0·2 mm long, by electrolysis of saturated $Hg(NO_3)_2$ soln. for 75 sec. at 30 mA. The sample (1 g) is treated with 8 ml of HBr (sp. gr. 1·48) and then dropwise with 2 ml of Br. The soln. is evaporated to dryness at 110° to 115°, more Br is added if necessary to obtain complete dissolution, and the soln. is re-evaporated. The residue is treated with 8 ml of HCl and 2 ml of HNO_3 and again evaporated at the same temp. The residue is then boiled with 10 ml of 0·1N-KOH and the soln. is diluted to 50 ml. An aliquot (20 ml) of the Sn-free soln. is transferred to the electrolysis cell and freed from O by a current of N, and preliminary electrolysis is carried out at −1·75 V for 30 min., with magnetic stirring except for the last 30 sec. The potential is then reduced linearly to −0·2 V and the current is continuously recorded. The depths of the anodic troughs corresponding to Zn (−1·17 V), Pb (−0·61 V) and Cu (−0·2 V) are measured and the results are converted into concn. by means of standards.

G. S. SMITH

4974. Gravimetric determination of small amounts of lead by means of a chromous salt solution. N. T. Vasina (Lugansk State Medical Inst.). *Zhur. Anal. Khim.*, 1961, **16** (2), 241–242.—Lead (1 to 4 mg) in soln. is quant. reduced to the metal by Cr^{2+} salts at pH 3·7, and may be filtered off and weighed. Arsenic and Zn do not interfere. Antimony is partly pptd., but can be removed. *Procedure for dilute soln.* (≃1 *mg per litre*).—Evaporate the soln. to 10 to 25 ml, cool, add 0·5N-$CrCl_2$ (10 ml) and set aside for 10 min. Filter off the ppt., wash it with ethanol, dry it at 105° and weigh. In the presence of Sb, wash the ppt. with 0·2N-$K_2Cr_2O_7$ in H_2SO_4 (1:4).

J. W. PRICE

4975. Determination of lead using an anion exchanger and sodium chloranilate. E. A. Wynne, R. D. Burdick and L. H. Fine (Chem. Mfg Div., Fisher Scientific Co., Fair Lawn, N.J., U.S.A.). *Anal. Chem.*, 1961, **33** (6), 807–808.—The use of the method described avoids the cleaning of glassware and purification of solvents required in conventional determinations. The Pb is converted into an anionic complex with N-HCl. The soln. is passed through a column of strongly basic anion-exchanger, which is then washed wth N-HCl. The lead complex is eluted with water and, after adjustment of the pH to 4·0, an aliquot of the soln. is treated with 1% aq. Na chloranilate and centrifuged. To the precipitate is added a 5% aq. soln. of EDTA (disodium salt) and the extinction is read after 10 min. at 530 mμ against a reagent blank. The calibration curve is linear over the range from 50 to 250 μg of Pb.

C. B. BAINES

4976. A field method for the detection of lead and zinc in ores. V. Marjanović-Krajovan, M. Ogrizek and K. Moskaliuk (Dept. of Anal. Chem., Fac. Technol., Zagreb Univ., Yugoslavia). *Kem. u Ind., Zagreb*, 1960, **9** (11), 261–263.—A rapid procedure was developed for application to sulphate, sulphide, oxide and carbonate ores.

Lead is detected with 0·5N-KI. A 2:3 mixture of a soln. of diethylaniline (0·5 g) in 200 ml of 50% H_3PO_4 and a soln. of $K_3Fe(CN)_6$ (2 g) in 100 ml of water is used as a spot-test reagent for Zn. A. L. GROCHOWSKI

4977. **Red lead for paints and jointing compounds.** British Standards Institution (2 Park Street, London, W.1). B.S. 217:1961. 15 pp.—Methods are specified for total Pb, total lead oxides, Pb_3O_4, matter volatile at 100° and matter sol. in water, in addition to physical methods for the evaluation of the compound under practical conditions.

4978. **Analysis of lead telluride with an accuracy to better than 0·1%.** K. L. Cheng (RCA Lab., Princeton, N.J., U.S.A.). *Anal. Chem.*, 1961, **33** (6), 761–764.—Lead is determined by complexing with EDTA, and back-titrating the excess of EDTA at pH 5·5 to 6·0 with standard $Pb(NO_3)_2$ soln., with methylthymol blue as indicator. Tellurium is determined by pptn. as tellurous acid in the presence of EDTA and weighing as TeO_2. The solubility of TeO_2 is shown to be at a minimum at pH 4·0 to 5·0 (0·1 mg of TeO_2 per 100 ml) and to increase appreciably at temp. > 40°. Citrate and tartrate are complexing agents for Te, and should be absent. G. S. ROBERTS

4979. **Polarography of quadrivalent titanium and zirconium in methanolic and aqueous alcoholic media.** P. G. Desideri and F. Pantani (Univ., Firenze, Italy). *Ric. Sci.*, 1960, **30** (12), 2000–2008.—In absolute methanol solution, Ti^{4+} are reduced reversibly with a dropping mercury electrode. The reaction is made irreversible by addition of H_2O. The ZrO^{2+} are not reduced either in methanolic or aq. methanolic media. The diffusion currents shown polarographically with $ZrOCl_2$ in methanol are due to the catalytic reduction of H^+ formed by hydrolysis of $ZrOCl_2$. The reduction of Ti^{4+} and the hydrolysis currents of $ZrOCl_2$ have been studied over a wide range of concn. of Cl^-, acidity and H_2O. The polarographic determination of Ti^{4+} should be carried out with methanol in the presence of $\gtrsim$0·5% (v/v) of H_2O and of Zr^{4+} in 20% (v/v) aq. methanol.
L. A. O'NEILL

4980. **Determination of micro amounts of sulphide sulphur in titanium and zirconium by means of the iodine - azide reaction.** L. V. Markova (Inst. of Gen. and Inorg. Chem., Acad. Sci., UkrSSR). *Zavod. Lab.*, 1961, **27** (4), 379–381.—The sample (0·1 to 1·5 g) of titanium, zirconium or steel in a reaction flask with an atmosphere of N is treated with 15 to 25 ml of an acid mixture (25 ml of $\simeq$2·1M-HCl and 3 to 4 ml of $\simeq$16M-HF) in the presence of 0·5 g of H_3BO_3 (to reduce attack on the glass). The H_2S is absorbed in 0·5% NaOH soln. An aliquot (0·5 to 2 ml) is treated with acetic acid and then with an acetate buffer soln. to give a pH of 5·3, and the S is determined photometrically by means of its action on an iodine - azide soln. [4M-KI (0·5 ml), 0·2M-NaN_3 (0·5 ml) and 0·0008M-iodine (0·5 ml)]. G. S. SMITH

4981. **Gravimetric determination of zirconium after precipitation with 1-hydroxycyclohexane-carboxylic acid.** I. P. Alimarin and Han-Hsi Hên (M. V. Lomonosov Moscow State Univ.). *Zhur. Anal. Khim.*, 1961, **16** (2), 162–165.—The reagent (**I**) (preparation described) gives ppt. of varying composition with Zr in dilute acid soln., all of which can be ignited to ZrO_2; Al, Fe, Bi, Th, U, V, Sn, Ti, Cr and the rare-earth elements do not interfere. *Determination in zirconium bronze*—Dissolve the sample in HNO_3 or HNO_3 - HCl, evaporate to dryness, dissolve the residue in HCl, dilute with 30 ml of H_2O and add 30 ml of 5% aq. **I** soln. (final acidity 0·4N). Filter off the ppt., wash it with 0·4N-HCl, ignite and weigh as ZrO_2. *Determination in minerals*—Fuse the sample with NaOH - Na_2O_2. Extract the melt with H_2O and filter off the $Zr(OH)_2$. Dissolve the ppt. in HCl (20 ml) by warming, filter off insoluble material, transfer the filtrate to a 100-ml flask and make up to vol. Precipitate the Zr from a suitable aliquot as described above. J. W. PRICE

4982. **Photometric determination of zirconium with xylenol orange.** V. F. Lukyanov and E. M. Knyazeva. *Zhur. Anal. Khim.*, 1961, **16** (2), 248–249.—Xylenol orange (**I**) in acid soln. forms a stable red complex with Zr having an absorption max. at 540 mμ (Cheng, *Anal. Abstr.*, 1959, **6**, 2916, 4716). *Procedure for ores*—Fuse 0·2 to 0·5 g of sample in a corundum crucible with 5 to 10 g of Na_2O_2. Extract the melt with H_2O (250 to 300 ml) and boil for 10 min. to destroy peroxide. Filter off and wash the ppt. with 5% NaOH soln., then dissolve it in hot 2N-HCl and dilute to 250 ml with the same acid. Evaporate a 5 to 10-ml aliquot with conc. HCl (5 ml) to a volume of $\simeq$ 1 ml, and transfer to a 50-ml flask. Add 0·5 ml of **I** soln. (0·8% aq.), make up to vol. with 0·5N-HCl and measure the extinction; Cr^{3+}, Bi^{3+}, Fe^{3+} and PO_4^{3-} interfere. J. W. PRICE

4983. **Fluorimetric determination of zirconium in the presence of titanium with quercetin.** A. P. Golovina, I. P. Alimarin and L. A. Tenyakova. *Vestn. Moskov. Univ., Ser. Khim.*, 1960, No. 3, 60–62.—Soln. of zirconium give with quercetin an orange-yellow fluorescence in u.v. light, but soln. of titanium do not fluoresce. *Procedure*—Dissolve 0·2 g of ore (containing Zr, Ti and Fe) in a platinum dish with a mixture of 4 ml of HF and 2 ml of H_2SO_4. Transfer the soln. to a 50-ml flask and dilute to vol. with H_2O. Place drops on a Whatman No. 1 filter-paper strip (30 cm × 3 cm) and separate the elements by descending paper chromatography with ethanol - HCl (4:1) for 8 hr. Treat the chromatogram with a 0·01% soln. of quercetin in ethanol and determine the Zr by comparing the fluorescence in u.v. light with that of standard spots of known Zr content. A. BURWOOD-SMITH

4984. **Absorptiometric determination of combined nitrogen in zirconium metal and Zircaloy.** U.K.A.E.A. (Production Group, Springfields, Lancs., England). U.K.A.E.A. Report PG15(S), 1961. 7 pp.—The sample (0·3 to 0·5 g) is dissolved in hot aq. H_2SO_4 (1:1) containing a little HF. The soln. is made alkaline by adding NaOH soln. and NH_3 is distilled off. Sodium phenoxide and NaClO soln. are added to the distillate and the absorption of the soln. is measured in a Spekker absorptiometer, Ilford 608 filters being used. The content of N is calculated by applying a factor determined by using known amounts of N. The coefficient of variation in the range 5 to 90 p.p.m. is $\simeq$3%. Full experimental details and a diagram of the distillation apparatus are given. G. J. HUNTER

4985. **Determination of silicon in zirconium and Zircaloy II.** E. Bowell and P. T. S. Sandon (U.K.A.E.A. Research Group, Riverside, Chatham, Kent). A.E.R.E. Analytical Method AERE-AM83,

1961. 7 pp.—The sample (≃0·5 g) is dissolved in 40% HF soln. and an excess of satd. H_3BO_3 soln. is added. The soln. is filtered, dil. H_2SO_4, ammonium molybdate and $SnCl_2$ soln. are added, and the absorption is measured in a Spekker absorptiometer; Ilford 608 filters are used. The content of Si is calculated from a factor determined by using known amounts of Si. The method is applicable to materials with contents of Si from 10 to 100 p.p.m., and the error, except for the lower concn., should be <5%.

G. J. HUNTER

4986. **Determination of thorium alpha-activity and of zirconium, cerium and rare-earth beta-activities in solutions containing aluminium.** C. G. Wallace (U.K.A.E.A. Research Group, C.37, Royal Arsenal, Woolwich, London, S.E.18). A.E.R.E. Analytical Method AERE-AM76, 1961. 12 pp.—Up to 80 mg of Al may be present in the sample, which is first treated with HF and $HClO_4$ in the presence of cerium, neodymium and zirconium carriers. The residue is treated with NaOH soln. to remove Al and is then dissolved in HNO_3 to which H_2O_2 is added. Zirconium and Th are then pptd. as iodates. Cerium is oxidised with $KBrO_3$ and pptd. as $Ce(IO_3)_4$ to separate it from other rare-earth metals. The Th is separated from Zr by co-pptn. of ThF_4 with CeF_3. After various purification steps the Ce and rare-earth metals are pptd. as oxalates and β-counted; the CeF_3-ThF_4 ppt. is α-counted; and the Zr is pptd. as mandelate, which is β-counted. The recovery of Th is ≃95%.

G. J. HUNTER

4987. **Determination of small amounts of gadolinium, samarium and europium in metallic thorium.** V. L. Levshin, E. Ya. Aranova and E. G. Baranova. *Trudy Komiss. Anal. Khim., Akad. Nauk SSSR*, 1960, **12**, 393–408; *Ref. Zhur., Khim.*, 1961, (3), Abstr. No. 3D147.—The method described is based on the preparation of a fluorescent compound by conversion of the thorium metal sample to sulphate and fusion with $Na_2B_4O_7$ and Na_2HPO_4. Samarium, Eu and Gd can act as activators in such phosphors, these elements being determined by reference to the intensity and spectral characteristics of the fluorescence of the preparation. Other impurities, *e.g.*, rare-earth elements, Fe, Mn, Cu and Ni, may interfere with the determination even when present at concn. as low as 50 p.p.m. A single determination takes 3 to 4 days; errors are within ±25%; the sensitivity for Gd is 10^{-6} %, and for Sm and Eu, 10^{-5} %.

B. S. COOPER

4988. **Determination of impurities in thorium and its compounds by a chemical - spectrographic method.** A. G. Karabash, Sh. I. Peizulaev, V. P. Usacheva, G. G. Morozova, V. M. Meshkova and V. L. Lobanova. *Zhur. Anal. Khim.*, 1961, **16** (2), 217–222.—Thorium is pptd. as oxalate or peroxide and the impurities remaining in soln. are concentrated by evaporation with $Be(NO_3)_2$ and determined spectrographically on the ignited residue. Two enrichment procedures are described. *Procedure* (*i*)—Dissolve the sample (3 g) in aqua regia in a quartz flask. Evaporate to dryness and add conc. HNO_3 (8 ml); dilute to 100 ml and to the warmed soln. add 2·64 g of oxalic acid as a 10% soln. Set aside for 4 hr. on a water bath, add a further 3·96 g of oxalic acid, set aside for 1 hr. on a water bath and then for 3 hr. at room temp. Decant the soln. into a quartz dish, add 100 mg of $Be(NO_3)_2$, evaporate to dryness and heat to 200° to 300°. Re-dissolve the residue in HNO_3 (3 ml) and evaporate to a syrup, then precipitate $Be(OH)_2$ by adding 25% aq. NH_3. Dry the ppt., heat it to 250° to 300°, and finally ignite it for 30 min. at 500°. *Procedure* (*ii*)—Dissolve the sample (0·25 g) in aqua regia as described above. Evaporate twice to dryness with HNO_3 and dissolve the residue in H_2O (3 ml) plus 5N-HNO_3 (1 ml). Dilute to 40 ml, add NH_4NO_3 (2 g), heat to 60° to 80° and add, slowly with stirring, 15% H_2O_2 soln. (10 ml). Set aside for 1 hr., centrifuge and wash the ppt. with 1% NH_4NO_3 soln. (3 × 3 ml). Dissolve the ppt. in 7N-HNO_3, evaporate, and re-precipitate as described above. Transfer the combined soln. and washings to a quartz dish, add 700 mg of $Be(NO_3)_2$ and continue as described above. Analyse spectrographically with a d.c. arc in a medium quartz spectrograph. By procedure (*i*) an enrichment coeff. of 20 to 30 is obtained and the following elements can be determined in a single spectrogram—Mg, Ca, Ba, Al, Ti, V, Cr, Mo, Mn, Fe, Co, Ni, Cu, Ag, Zn, Cd, Sn and Sb. By procedure (*ii*) an enrichment coeff. of 2·5 to 5 is obtained and Mg, Ca, Ba, Al, Cr, Mo, Mn, Fe, Co, Ni, Cu, Ag, Zn, Cd and Pb can be determined.

J. W. PRICE

4989. **The determination of combined nitrogen in rocks and minerals.** F. Wlotzka (Sedimentpetrographisch. Inst., Univ. Göttingen, Germany). *Geochim. et Cosmoch. Acta*, 1961, **24** (1–2), 106–154 (in German).—Two methods are described. (*i*)—For the determination of total combined N, rock is decomposed by fusion with NaOH in a closed system, and the melt is dissolved in water. A part of the existing NH_3 is oxidised to NO_3^- and it is thus impossible to determine NH_3 and NO_3^- separately. The NO_3^- are reduced by Devarda's alloy to NH_3, which is distilled off with the primary NH_3, absorbed in standard acid and determined colorimetrically by the Nessler method. (*ii*)—For the determination of ammonia-N and nitrate-N separately, the sample is decomposed with HF at 90° in a closed polyethylene bottle, the acid is neutralised with NaOH and after distillation the determination of NH_3 is carried out as described above. This is followed by determination of NO_3^- as NH_3 after reduction by Devarda's alloy. (*Cf.* Stevenson, *Anal. Abstr.*, 1961, **8**, 2443).

R. A. HOWIE

4990. **Determination of microgram amounts of phosphates. A new general technique.** E. A. Wynne, R. D. Burdick and L. H. Fine (Res. and Dev. Lab., Fisher Scientific Co., Fair Lawn, N.J., U.S.A.). *Microchem. J.*, 1961, **5** (2), 185–191.—*Procedure*—Pass the PO_4^{3-} soln. (up to 50 mg of PO_4^{3-}) through a cation-exchanger (H^+ form) and then through an anion-exchanger (OH^- form). Elute interfering F^- with 0·25N-NaOH and then the PO_4^{3-} with N-NaOH. Again pass this soln. through the cation-exchanger. Use half the percolate (≃ 150 ml) for analysis. Adjust the pH to ≃ 4 with 10% HCl, then add successively H_2O (to a total vol. of 165 ml), acetate buffer (pH 4·63) (25 ml), 2-butoxyethanol (60 ml) and La chloranilate (0·3 g). Shake at 5-min. intervals and filter after 30 min. Read the extinction at 530 mμ or at 330 mμ for smaller amounts (fractions of a p.p.m.). The recovery was > 99%.

G. RUSSELL

4991. **Determination of arsenic in the presence of antimony and the behaviour of the antimony.** H. Ploum. *Arch. Eisenhüttenw.*, 1961, **32** (2), 71-

Lead is detected with 0·5N-KI. A 2:3 mixture of a soln. of diethylaniline (0·5 g) in 200 ml of 50% H_3PO_4 and a soln. of $K_3Fe(CN)_6$ (2 g) in 100 ml of water is used as a spot-test reagent for Zn. A. L. GROCHOWSKI

4977. **Red lead for paints and jointing compounds.** British Standards Institution (2 Park Street, London, W.1). B.S. 217:1961. 15 pp.—Methods are specified for total Pb, total lead oxides, Pb_3O_4, matter volatile at 100° and matter sol. in water, in addition to physical methods for the evaluation of the compound under practical conditions.

4978. **Analysis of lead telluride with an accuracy to better than 0·1%.** K. L. Cheng (RCA Lab., Princeton, N.J., U.S.A.). *Anal. Chem.*, 1961, **33** (6), 761–764.—Lead is determined by complexing with EDTA, and back-titrating the excess of EDTA at pH 5·5 to 6·0 with standard $Pb(NO_3)_2$ soln., with methylthymol blue as indicator. Tellurium is determined by pptn. as tellurous acid in the presence of EDTA and weighing as TeO_2. The solubility of TeO_2 is shown to be at a minimum at pH 4·0 to 5·0 (0·1 mg of TeO_2 per 100 ml) and to increase appreciably at temp. > 40°. Citrate and tartrate are complexing agents for Te, and should be absent. G. S. ROBERTS

4979. **Polarography of quadrivalent titanium and zirconium in methanolic and aqueous alcoholic media.** P. G. Desideri and F. Pantani (Univ., Firenze, Italy). *Ric. Sci.*, 1960, **30** (12), 2000–2008.—In absolute methanol solution, Ti^{4+} are reduced reversibly with a dropping mercury electrode. The reaction is made irreversible by addition of H_2O. The ZrO^{2+} are not reduced either in methanolic or aq. methanolic media. The diffusion currents shown polarographically with $ZrOCl_2$ in methanol are due to the catalytic reduction of H^+ formed by hydrolysis of $ZrOCl_2$. The reduction of Ti^{4+} and the hydrolysis currents of $ZrOCl_2$ have been studied over a wide range of concn. of Cl^-, acidity and H_2O. The polarographic determination of Ti^{4+} should be carried out with methanol in the presence of $\gtrsim$0·5% (v/v) of H_2O and of Zr^{4+} in 20% (v/v) aq. methanol.
L. A. O'NEILL

4980. **Determination of micro amounts of sulphide sulphur in titanium and zirconium by means of the iodine - azide reaction.** L. V. Markova (Inst. of Gen. and Inorg. Chem., Acad. Sci., UkrSSR). *Zavod. Lab.*, 1961, **27** (4), 379–381.—The sample (0·1 to 1·5 g) of titanium, zirconium or steel in a reaction flask with an atmosphere of N is treated with 15 to 25 ml of an acid mixture (25 ml of $\simeq$2·1M-HCl and 3 to 4 ml of $\simeq$16M-HF) in the presence of 0·5 g of H_3BO_3 (to reduce attack on the glass). The H_2S is absorbed in 0·5% NaOH soln. An aliquot (0·5 to 2 ml) is treated with acetic acid and then with an acetate buffer soln. to give a pH of 5·3, and the S is determined photometrically by means of its action on an iodine - azide soln. [4M-KI (0·5 ml), 0·2M-NaN_3 (0·5 ml) and 0·0008M-iodine (0·5 ml)]. G. S. SMITH

4981. **Gravimetric determination of zirconium after precipitation with 1-hydroxycyclohexane-carboxylic acid.** I. P. Alimarin and Han-Hsi Hên (M. V. Lomonosov Moscow State Univ.). *Zhur. Anal. Khim.*, 1961, **16** (2), 162–165.—The reagent (**I**) (preparation described) gives ppt. of varying composition with Zr in dilute acid soln., all of which can be ignited to ZrO_2; Al, Fe, Bi, Th, U, V, Sn, Ti, Cr and the rare-earth elements do not interfere. *Determination in zirconium bronze*—Dissolve the sample in HNO_3 or HNO_3 - HCl, evaporate to dryness, dissolve the residue in HCl, dilute with 30 ml of H_2O and add 30 ml of 5% aq. **I** soln. (final acidity 0·4N). Filter off the ppt., wash it with 0·4N-HCl, ignite and weigh as ZrO_2. *Determination in minerals*—Fuse the sample with NaOH - Na_2O_2. Extract the melt with H_2O and filter off the $Zr(OH)_4$. Dissolve the ppt. in HCl (20 ml) by warming, filter off insoluble material, transfer the filtrate to a 100-ml flask and make up to vol. Precipitate the Zr from a suitable aliquot as described above. J. W. PRICE

4982. **Photometric determination of zirconium with xylenol orange.** V. F. Lukyanov and E. M. Knyazeva. *Zhur. Anal. Khim.*, 1961, **16** (2), 248–249.—Xylenol orange (**I**) in acid soln. forms a stable red complex with Zr having an absorption max. at 540 mμ (Cheng, *Anal. Abstr.*, 1959, **6**, 2916, 4716). *Procedure for ores*—Fuse 0·2 to 0·5 g of sample in a corundum crucible with 5 to 10 g of Na_2O_2. Extract the melt with H_2O (250 to 300 ml) and boil for 10 min. to destroy peroxide. Filter off and wash the ppt. with 5% NaOH soln., then dissolve it in hot 2N-HCl and dilute to 250 ml with the same acid. Evaporate a 5 to 10-ml aliquot with conc. HCl (5 ml) to a volume of $\simeq$ 1 ml, and transfer to a 50-ml flask. Add 0·5 ml of **I** soln. (0·8% aq.), make up to vol. with 0·5N-HCl and measure the extinction; Cr^{3+}, Bi^{3+}, Fe^{3+} and PO_4^{3-} interfere. J. W. PRICE

4983. **Fluorimetric determination of zirconium in the presence of titanium with quercetin.** A. P. Golovina, I. P. Alimarin and L. A. Tenyakova. *Vestn. Moskov. Univ., Ser. Khim.*, 1960, No. 3, 60–62.—Soln. of zirconium give with quercetin an orange-yellow fluorescence in u.v. light, but soln. of titanium do not fluoresce. *Procedure*—Dissolve 0·2 g of ore (containing Zr, Ti and Fe) in a platinum dish with a mixture of 4 ml of HF and 2 ml of H_2SO_4. Transfer the soln. to a 50-ml flask and dilute to vol. with H_2O. Place drops on a Whatman No. 1 filter-paper strip (30 cm × 3 cm) and separate the elements by descending paper chromatography with ethanol - HCl (4:1) for 8 hr. Treat the chromatogram with a 0·01% soln. of quercetin in ethanol and determine the Zr by comparing the fluorescence in u.v. light with that of standard spots of known Zr content. A. BURWOOD-SMITH

4984. **Absorptiometric determination of combined nitrogen in zirconium metal and Zircaloy.** U.K.A.E.A. (Production Group, Springfields, Lancs., England). U.K.A.E.A. Report PG15(S), 1961. 7 pp.—The sample (0·3 to 0·5 g) is dissolved in hot aq. H_2SO_4 (1:1) containing a little HF. The soln. is made alkaline by adding NaOH soln. and NH_3 is distilled off. Sodium phenoxide and NaClO soln. are added to the distillate and the absorption of the soln. is measured in a Spekker absorptiometer, Ilford 608 filters being used. The content of N is calculated by applying a factor determined by using known amounts of N. The coefficient of variation in the range 5 to 90 p.p.m. is $\simeq$3%. Full experimental details and a diagram of the distillation apparatus are given. G. J. HUNTER

4985. **Determination of silicon in zirconium and Zircaloy II.** E. Bowell and P. T. S. Sandon (U.K.A.E.A. Research Group, Riverside, Chatham, Kent). A.E.R.E. Analytical Method AERE-AM83,

1961. 7 pp.—The sample (≃0·5 g) is dissolved in 40% HF soln. and an excess of satd. H_3BO_3 soln. is added. The soln. is filtered, dil. H_2SO_4, ammonium molybdate and $SnCl_2$ soln. are added, and the absorption is measured in a Spekker absorptiometer; Ilford 608 filters are used. The content of Si is calculated from a factor determined by using known amounts of Si. The method is applicable to materials with contents of Si from 10 to 100 p.p.m., and the error, except for the lower concn., should be <5%.

G. J. HUNTER

4986. **Determination of thorium alpha-activity and of zirconium, cerium and rare-earth beta-activities in solutions containing aluminium.** C. G. Wallace (U.K.A.E.A. Research Group, C.37, Royal Arsenal, Woolwich, London, S.E.18). A.E.R.E. Analytical Method AERE-AM76, 1961. 12 pp.—Up to 80 mg of Al may be present in the sample, which is first treated with HF and $HClO_4$ in the presence of cerium, neodymium and zirconium carriers. The residue is treated with NaOH soln. to remove Al and is then dissolved in HNO_3 to which H_2O_2 is added. Zirconium and Th are then pptd. as iodates. Cerium is oxidised with $KBrO_3$ and pptd. as $Ce(IO_3)_4$ to separate it from other rare-earth metals. The Th is separated from Zr by co-pptn. of ThF_4 with CeF_3. After various purification steps the Ce and rare-earth metals are pptd. as oxalates and β-counted; the CeF_3-ThF_4 ppt. is α-counted; and the Zr is pptd. as mandelate, which is β-counted. The recovery of Th is ≃95%.

G. J. HUNTER

4987. **Determination of small amounts of gadolinium, samarium and europium in metallic thorium.** V. L. Levshin, E. Ya. Aranova and E. G. Baranova. *Trudy Komiss. Anal. Khim., Akad. Nauk SSSR*, 1960, **12**, 393–408; *Ref. Zhur., Khim*, 1961, (3), Abstr. No. 3D147.—The method described is based on the preparation of a fluorescent compound by conversion of the thorium metal sample to sulphate and fusion with $Na_2B_4O_7$ and Na_2HPO_4. Samarium, Eu and Gd can act as activators in such phosphors, these elements being determined by reference to the intensity and spectral characteristics of the fluorescence of the preparation. Other impurities, *e.g.*, rare-earth elements, Fe, Mn, Cu and Ni, may interfere with the determination even when present at concn. as low as 50 p.p.m. A single determination takes 3 to 4 days; errors are within ±25%; the sensitivity for Gd is 10^{-5} %, and for Sm and Eu, 10^{-4} %.

B. S. COOGER

4988. **Determination of impurities in thorium and its compounds by a chemical - spectrographic method.** A. G. Karabash, Sh. I. Peizulaev, V. P. Usacheva, G. G. Morozova, V. M. Meshkova and V. L. Lobanova. *Zhur. Anal. Khim.*, 1961, **16** (2), 217–222.—Thorium is pptd. as oxalate or peroxide and the impurities remaining in soln. are concentrated by evaporation with $Be(NO_3)_2$ and determined spectrographically on the ignited residue. Two enrichment procedures are described. *Procedure* (*i*)—Dissolve the sample (3 g) in aqua regia in a quartz flask. Evaporate to dryness and add conc. HNO_3 (8 ml); dilute to 100 ml and to the warmed soln. add 2·64 g of oxalic acid as a 10% soln. Set aside for 4 hr. on a water bath, add a further 3·96 g of oxalic acid, set aside for 1 hr. on a water bath and then for 3 hr. at room temp. Decant the soln. into a quartz dish, add 100 mg of $Be(NO_3)_2$, evaporate to dryness and heat to 200° to 300°. Re-dissolve the residue in HNO_3 (3 ml) and evaporate to a syrup, then precipitate $Be(OH)_2$ by adding 25% aq. NH_3. Dry the ppt., heat it to 250° to 300°, and finally ignite it for 30 min. at 500°. *Procedure* (*ii*)—Dissolve the sample (0·25 g) in aqua regia as described above. Evaporate twice to dryness with HNO_3 and dissolve the residue in H_2O (3 ml) plus 5N-HNO_3 (1 ml). Dilute to 40 ml, add NH_4NO_3 (2 g), heat to 60° to 80° and add, slowly with stirring, 15% H_2O_2 soln. (10 ml). Set aside for 1 hr., centrifuge and wash the ppt. with 1% NH_4NO_3 soln. (3 × 3 ml). Dissolve the ppt. in 7N-HNO_3, evaporate, and re-precipitate as described above. Transfer the combined soln. and washings to a quartz dish, add 700 mg of $Be(NO_3)_2$ and continue as described above. Analyse spectrographically with a d.c. arc in a medium quartz spectrograph. By procedure (*i*) an enrichment coeff. of 20 to 30 is obtained and the following elements can be determined in a single spectrogram—Mg, Ca, Ba, Al, Ti, V, Cr, Mo, Mn, Fe, Co, Ni, Cu, Ag, Zn, Cd, Sn and Sb. By procedure (*ii*) an enrichment coeff. of 2·5 to 5 is obtained and Mg, Ca, Ba, Al, Cr, Mo, Mn, Fe, Co, Ni, Cu, Ag, Zn, Cd and Pb can be determined.

J. W. PRICE

4989. **The determination of combined nitrogen in rocks and minerals.** F. Wlotzka (Sedimentpetrographisch. Inst., Univ. Göttingen, Germany). *Geochim. et Cosmoch. Acta*, 1961, **24** (1–2), 106–154 (in German).—Two methods are described. (*i*)—For the determination of total combined N, rock is decomposed by fusion with NaOH in a closed system, and the melt is dissolved in water. A part of the existing NH_3 is oxidised to NO_3^- and it is thus impossible to determine NH_3 and NO_3^- separately. The NO_3^- are reduced by Devarda's alloy to NH_3, which is distilled off with the primary NH_3, absorbed in standard acid and determined colorimetrically by the Nessler method. (*ii*)—For the determination of ammonia-N and nitrate-N separately, the sample is decomposed with HF at 90° in a closed polyethylene bottle, the acid is neutralised with NaOH and after distillation the determination of NH_3 is carried out as described above. This is followed by determination of NO_3^- as NH_3 after reduction by Devarda's alloy. (*Cf.* Stevenson, *Anal. Abstr.*, 1961, **8**, 2443).

R. A. HOWIE

4990. **Determination of microgram amounts of phosphates. A new general technique.** E. A. Wynne, R. D. Burdick and L. H. Fine (Res. and Dev. Lab., Fisher Scientific Co., Fair Lawn, N.J., U.S.A.). *Microchem. J.*, 1961, **5** (2), 185–191.—*Procedure*—Pass the PO_4^{3-} soln. (up to 50 mg of PO_4^{3-}) through a cation-exchanger (H^+ form) and then through an anion-exchanger (OH^- form). Elute interfering F^- with 0·25N-NaOH and then the PO_4^{3-} with N-NaOH. Again pass this soln. through the cation-exchanger. Use half the percolate (≃ 150 ml) for analysis. Adjust the pH to ≃ 4 with 10% HCl, then add successively H_2O (to a total vol. of 165 ml), acetate buffer (pH 4·63) (25 ml), 2-butoxyethanol (60 ml) and La chloranilate (0·3 g). Shake at 5-min. intervals and filter after 30 min. Read the extinction at 530 mμ or at 330 mμ for smaller amounts (fractions of a p.p.m.). The recovery was > 99%.

G. RUSSELL

4991. **Determination of arsenic in the presence of antimony and the behaviour of the antimony.** H. Ploum. *Arch. Eisenhüttenw.*, 1961, **32** (2), 71-

76.—A series of tests were performed in the study of the behaviour of Sb during the distillation of $AsCl_3$. A lowering of the final temp. from 135° to 120° by using 5 times as much HCl markedly reduces the distillation of $SbCl_3$. A second distillation reduces the $SbCl_3$ to a negligible amount, if the amount of Sb in the boiling flask does not exceed 80 mg. On the basis of these tests a method was developed which, with certain variations, was applied to the accurate determination of As in iron, steel and ferrous alloys and in ores and slag.
M. L.

4992. **Separation of arsenic, antimony and tin by fractional distillation.** P. De Bruyne and J. Hoste (Anal. Chem. Lab., Univ. Ghent, Belgium). *Bull. Soc. Chim. Belg.*, 1961, **70** (5–6), 221–230.—Apparatus and procedure are described for the quant. separation of As, Sb and Sn (0·01 to 0·8 mg of each) from nodular cast-iron by successive fractional distillations of the three halides in a stream of CO_2. The As is separated first by distillation from 11N-HCl at 109° for ≃40 min.; 85% H_3PO_4 soln. is then added to form a stable complex with Sn and the Sb is distilled from 11N-HCl at 155° to 165° for ≃110 min. Finally, the Sn is liberated from its complex by the addition of a mixture of 11N-HCl and aq. HBr (sp. gr. 1·377) (3:1) and distilled at 140° for ≃75 min. The prolonged distillation ensures negligible contamination by Fe. The results reported were obtained on mixtures of As_2O_3, Sb_2O_3 and metallic tin with iron powder (150 to 200 mesh) compressed into pellets (0·2 to 0·8 g) and irradiated to form ^{76}As, ^{122}Sb and ^{121}Sn. The distillation yields were calculated from the radiometric analyses. This method can be used to determine trace amounts of As, Sb and Sn in cast iron.
W. J. Baker

4993. **Determination of niobium-95 in reactor fuel processing and effluent treatment plant solutions.** U.K.A.E.A. (Production Group, Technical Branch, Windscale, Cumb.). U.K.A.E.A. Report PG211(W), 1961. 9 pp.—Full experimental details are given. After pptn. of the rare-earth-metal fluorides, ^{95}Nb and added carrier are pptd. with aq. NH_3 from a soln. containing zirconium holdback carrier. The ppt. is dissolved in HNO_3 and oxalic acid soln. and re-pptd. by heating with $KBrO_3$. The Nb_2O_5 is dissolved in $NH_4F.HF$ soln. and re-pptd. with aq. NH_3. The Nb_2O_5 is finally ignited and γ-counted.
G. J. Hunter

4994. **Determination of small amounts of niobium in ores containing titanium, tungsten, molybdenum and chromium.** V. M. Dorosh (Irkutsk Sci. Res. Inst. Rare Metals). *Zhur. Anal. Khim.*, 1961, **16** (2), 250–252.—Niobium is separated from Cu, Ni, Mo, Sn, Si, Al, Ti, Zr and Fe by alkaline fusion followed by co-pptn. with MnO_2, and determined photometrically with thiocyanate. *Procedure*—Fuse 0·1 to 0·2 g of the sample with 5 to 10 g of NaOH in a nickel or iron crucible. Extract the melt with H_2O and boil for 2 to 3 min. Filter off and wash the ppt. three to five times with 2% NaOH soln. Dissolve it in H_2SO_4 (1:125) containing a few drops of H_2O_2, add H_2SO_4 (1:2) (6 to 8 ml) and boil to destroy H_2O_2. Add N-$MnSO_4$ (1 ml) and $(NH_4)_2S_2O_8$ (2 g) and dilute to 200 ml. Boil for 10 min., filter off and wash the ppt. with H_2SO_4 (1:125). Fuse the ppt. with $NaHSO_4$ (1·5 to 3 g), dissolve the melt in 15% tartaric acid soln. (25 ml) and dilute to 50 ml. Determine the Nb as described by Freund and Levitt (*Anal. Chem.*, 1951, **23**, 1813) or Alimarin and Podval'naya (*Zhur. Anal. Khim.*, 1946, **1**, 30).
J. W. Price

4995. **Spectrographic determination of small amounts of aluminium and silicon in niobium pentoxide.** T. M. Moroshkina and G. F. Malinin (A. A. Zhdanov Leningrad State Univ.). *Zhur. Anal. Khim.*, 1961, **16** (2), 245–247.—By mixing the sample (20 to 40 mg) with half its weight of AgCl, Al down to 0·001% and Si down to 0·005% are preferentially volatilised, and can be determined with an accuracy of ±12%. The Al line at 3082·16 A is compared with the Nb line at 3097·12 A, and the Si line at 2881·58 A with the Nb line at 2884·97 A.
J. W. Price

4996. **Tentative method for estimating sulphides in shale.** S. Neglia (Agip Mineraria, Serv. Geochim., Milano, Italy). *Clay Min. Bull.*, 1961, **4** (25), 243–245.—The method is based on the oxidation of reducing substances by dil. HNO_2 (32·5% w/w), the absorption of the nitrous oxides in 5% KOH soln., oxidation with 0·2N-$KMnO_4$, and titration of the excess of $KMnO_4$ with 0·2N-Na oxalate.
R. A. Howie

4997. **Determination of sulphate in ferrous sulphamate feed solutions (EDTA titration method).** U.K.A.E.A. (Production Group, Technical Branch, Windscale, Cumberland). U.K.A.E.A. Report PG230(W), 1961. 4 pp.—Cations are removed from the soln. by passing it through a column of Zeo-Karb 225. An excess of a standard soln. of $BaCl_2$ is added and, after adjustment of the pH, the excess of Ba is determined by titration with EDTA (disodium salt), Solochrome black T (C.I. Mordant Black 11) being used as an indicator; hence the concn. of SO_4^{2-} in the sample is calculated.
G. J. Hunter

4998. **Separation and determination of small amounts of selenium and tellurium.** A. I. Ivankova and I. A. Blyum (Kazakh Sci. Res. Inst. of Mineral Raw Materials). *Zavod. Lab.*, 1961, **27** (4), 371–377.—In the preliminary treatment and concentration of small amounts (<10^{-4} %) of Se and Te, the sample (>2 g) is treated with 35 to 40 ml of conc. HNO_3 and, in the case of sulphide ores, with 0·05 to 0·1 g of KI or 0·1 to 0·2 g of $KClO_3$, and set aside for 24 hr. The soln. is evaporated to small vol., 10 ml of H_2SO_4 (1:1) is added, evaporation is continued to fuming, 5 to 10 ml of water is added, and the evaporation to fuming is repeated. The residue is boiled with 25 to 30 ml of water, the soln. is filtered, the insol. matter is washed with hot water, and the filtrate (>50 ml) is treated with an equal vol. of conc. HCl, 1 ml of As_2O_3 soln. (1 mg per ml) and 0·1 g of $CuSO_4$, together with some paper pulp. The soln. at 80° to 90° is treated while stirring with small amounts of NaH_2PO_2 until Fe^{3+} have been reduced and then with 1 to 2 g in excess. The soln. is boiled for 10 min. and set aside for <2 hr. The ppt. is collected, washed with hot 3% HCl (4 or 5 times) and then with water (3 or 4 times), and dissolved in a hot mixture of 2 to 3 drops of HCl and 5 ml of HNO_3, the filter being washed with hot water. The filtrate is treated with exactly 2 ml of H_2SO_4 (1:1) and evaporated to fuming, followed by addition of water and re-evaporation to fuming. The cooled residue is diluted to 10 ml. *Determination of Se*—An aliquot (3 ml) is treated with 2 ml

of 2·5N-formic acid and 3 ml of 0·1M-EDTA (disodium salt) and diluted to 35 ml. Aq. NH_3 (1:1) to give a pH of between 2 and 3 is added followed by 2 ml of 3,3'-diaminobenzidine. After 30 to 50 min. the pH is brought to between 6 and 7 and the soln. (≃50 ml) is extracted with 10 ml of toluene. The extinction of the extract at 410 mμ is measured after 15 to 20 min. The content is found from a calibration curve covering between 0 and 50 μg of Se. *Determination of Te*—An aliquot (3 ml) is mixed with 5·8 ml of H_2SO_4 (1:1), 0·6 ml of 3·5N-HBr, 0·6 ml of 0·1% butylrhodamine B soln. and 0·05 to 0·1 g of ascorbic acid. The soln. is shaken with 5 ml of benzene for 30 sec. The extinction of the extract at 530 mμ is measured after 20 min.

G. S. Smith

4999. **Thiomalic [mercaptosuccinic] acid as a reagent for the photometric determination of molybdenum.** A. I. Busev and Fan Chang (M. V. Lomonosov Moscow State Univ.). *Zhur. Anal. Khim.*, 1961, **16** (2), 171–179.—The reagent (**I**) forms a yellow complex with Mo^{VI} in acid soln., and Beer's law is obeyed over the range of 1 to 40 μg of Mo per ml. *Procedure for alloys containing Ti or Zr*—Dissolve 0·25 g of the sample in HCl (1:1) (50 ml) and oxidise with HNO_3. Dilute to 100 ml and take an aliquot containing ≯1 mg of Mo. Adjust the acid concn. to 0·5M and add 7·5 ml of 0·5% aq. **I** soln. Dilute to 25 ml and measure the extinction at 365 mμ after 20 min. In the presence of Ba, Mg, Ca, Ni, Zr, Mn, U, Be, Cu or Cr, which interfere, adjust the final pH to 3·6 and measure the extinction at 400 mμ; EDTA and citric, tartaric and oxalic acids must be absent. *Procedure for high-alloyed steel*—Dissolve 1 g of sample in HNO_3 (1:1) (20 ml) plus H_2SO_4 (1:1) (20 ml) and evaporate to fuming. Cool, add 50 to 80 ml of H_2O, heat to dissolve, and filter. Dilute the filtrate to 200 ml and transfer a suitable aliquot to a 50-ml flask. Add 5 to 10 ml of acetate buffer (pH 3·6) and 3 ml of **I** soln., dilute to volume and measure the extinction as described above.

J. W. Price

5000. **Electrometric titration of molybdic acid.** S. P. Moulik (Chem. Dept., Univ. Coll. of Sci., Calcutta, India). *Sci. & Cult.*, 1961, **27** (1), 45–46.—Travers and Malapride (*Bull. Soc. Chim.*, 1926, **39**, 1408) obtained two inflections in the titration curve when titrating aq. soln. of hydrated yellow molybdenum trioxide with KOH and aq. NH_3, and Britton and German (*J. Chem. Soc.*, 1930, 2154) obtained only one inflection when titrating soln. of sodium molybdate with HCl. The basicity of molybdic acid has therefore been re-investigated. A soln. of sodium molybdate (≃0·1N) was passed through a 30-cm column of a strong hydrogen-ion-exchange resin, to yield a soln. of molybdic acid of pH 2·26. When this soln. was titrated electrometrically with NaOH soln. (glass electrode) the curve of pH *vs.* vol. of titrant showed two inflections, one at ≃3 ml and the other at ≃ 10 ml.

R. E. E.

5001. **Routine quantitative analysis [of tungsten] by X-ray diffractometry.** P. Gadó (Telecommun. Ind. Res. Inst., Budapest). *Magyar Kém. Foly.*, 1961, **67** (5), 189–193.—The quant. analysis of powders containing α- and β-tungsten and tungsten dioxide (any two or all three being present together in a given mixture) was carried out by means of a recording X-ray diffractometer with a Geiger-Müller tube as radiation detector; 30 to 45 min. was required for the analysis and the error was < ±3%.

B. Vaszko

5002. **Improvements in or relating to a method of testing for uranium and apparatus therefor.** United Kingdom Atomic Energy Authority [Inventors: H. Mason, J. R. Sanderson and T. Mason]. Brit. Pat. 868,137, date appl. 31.10.57.—An org. solvent is tested for the presence of U by washing it with an aq. solution of a colour reagent for U (ammonium thioglycollate) to bring all the U into the aq. solution. Part of the coloured aq. solution is treated with a reagent that discharges the colour due to U, and the difference between the colour intensities before and after this treatment is measured.

J. M. Jacobs

5003. **3,6-Bis-(4-chloro-2-phosphonophenylazo)-4,5-dihydroxynaphthalene-2,7-disulphonic acid (chlorphosphonazo III)—a new reagent for the photometric determination of uranium.** A. A. Nemodruk, Yu. P. Novikov, A. M. Lukin and I. D. Kalinina (V. I. Vernadsky Inst. Geochem. and Anal. Chem., Acad. Sci. USSR, Moscow). *Zhur. Anal. Khim.*, 1961, **16** (2), 180–184.—The reagent (**I**) (preparation described) forms a 1:1 complex with U^{VI} having an absorption maximum at 670 mμ and a molecular extinction coeff. of 78,600. Fluorides, phosphates, oxalates, sulphates, tartrates and EDTA do not interfere, but small amounts of Th, Zr, Hf, Ce^{IV} and Ti^{IV} do. The interference of Zr and Hf can be masked by the addition of fluoride and that of Ti by adding H_2O_2. Larger amounts of a number of other elements interfere. *Procedure for alloys of uranium with zirconium*—Dissolve 0·1 g of sample in HCl (1:1) (10 ml) plus 0·5 g of NH_4F. Add 0·1M-Na_2SiO_3 (20 ml) and dilute to 250 ml. To a 1-ml aliquot add 5 ml of buffer soln. (10 mg of Na chloroacetate plus 20 g of chloroacetic acid per litre), 2·5 ml of 0·1M-Na_2SiO_3, 12·5 ml of 0·1M-NH_4F and 2 ml of 0·001M-**I**. Dilute to 100 ml and measure the extinction at 670 mμ.

J. W. Price

5004. **Determination of traces of uranium in minerals and natural water.** V. B. Spivakovskii, V. A. Zimina and L. S. Gavrilyuk (T. G. Shevchenko Kiev State Univ.). *Zavod. Lab.*, 1961, **27** (4), 390–391.—For minerals the sample (1 to 2 g) is heated during periodic grinding with a mixture of 9 ml of HNO_3 and 3 ml of HCl. After evaporation to dryness the residue is moistened with HNO_3 and evaporated nearly to dryness. The cooled residue is stirred with ethyl ether (4 × 3 ml), the ether extracts being poured into a porcelain dish. After addition of 1 or 2 drops of 0·1M-$Al(NO_3)_3$ the ether is evaporated off and the residue is gently ignited. After addition of 2 drops of HNO_3 and evaporation to dryness the residue is moistened with water and the U is extracted with ethyl ether (2-ml portions). The extracts are treated with 1 or 2 drops of water and 0·20 g of NaF, the ether and water are evaporated off and the residue is fused in a platinum-wire loop or small spoon. The fluorescence is compared with that of a series of standards.

G. S. Smith

5005. **Determination of particulate uranium on atmospheric dust adhesion plates (fluorimetric method).** U.K.A.E.A. (Production Group, Capenhurst, near Chester). U.K.A.E.A. Report PG224(CA), 1961. 7 pp.—Light petroleum is used

to wash the dust from the greased plates and is then evaporated. The residue is wet-oxidised with HNO_3 and H_2SO_4 and is finally obtained as a soln. in dilute HNO_3. An aliquot of the soln. is evaporated in a platinum capsule and then fused at 1000° with NaF - $NaHCO_3$ (1:9) for 70 sec. The fluorescence of the cooled melt is measured with a fluorimeter (A.E.R.E. Type 1080A), calibrated with known amounts of U. From 0·01 to 20 μg of U may be determined and the coefficient of variation is estimated to be 20%. Full experimental details are given. G. J. HUNTER

5006. **Influence of vanadium on the determination of uranium by dithionite - phosphate titrimetric and photometric methods.** V. M. Brodskaya, G. A. Lanskoi and V. G. Sochevanov. *Zhur. Anal. Khim.*, 1961, **16** (2), 185–190.—Methods of determination of U based on the pptn. of U^{IV} phosphate give high results in the presence of V, owing to the adsorption of V^{IV} compounds. These compounds are oxidised by iodine to V^{V} in soln. of pH >4, and if the phosphate ppt. are washed with an aq. iodine soln. of pH ≃5·5, the V is removed and correct results are obtained.

J. W. PRICE

5007. **Inorganic thin-layer chromatography. II. Identification of uranium(VI) and gallium(III) in mixtures with cations.** H. Seiler and M. Seiler (Inst. f. anorg. Chem., Univ. Basel). *Helv. Chim. Acta*, 1961, **44** (4), 939–941 (in German).—The thin-layer method was applied to the chromatographic identification and separation of UO_2^{2+} from Fe, Cu, Co, Ni, Cr, Al and Th, and of Ga^{3+} from Al^{3+}. M. SULZBACHER

5008. **Rapid spectrophotometric determination of vanadium and molybdenum in uranium materials.** P. R. Kuehn, O. H. Howard and C. W. Weber (Gaseous Diffusion Plant, Union Carbide Nuclear Co., Oak Ridge, Tenn., U.S.A.). *Anal. Chem.*, 1961, **33** (6), 740–744.—Vanadium and Mo are extracted into n-hexanol as their benzohydroxamate complexes, and the extinction due to V is measured. The Mo is then reduced to form its thiocyanate complex, the extinction of which is measured. *Procedure*—The sample (1 to 7 g of UF_6 hydrolysed in 50 ml of H_2O, or a suitable aliquot of uranium soln.) is oxidised with 1 ml of 0·2% $K_2Cr_2O_7$ soln., and the pH is adjusted to 2 ± 0·3. If F^- are present, 3 g of H_3BO_3 must first be added. The soln. is diluted to ≃100 ml and extracted with 20 ml of n-hexanol and 2 ml of 0·2M-aq. benzohydroxamic acid (**I**) (pH 5). The organic layer is shaken with a further 2 ml of **I** and then with 10 ml of phosphate soln. [20% aq. $(NH_4)_2HPO_4$ at pH 1·5] to remove Fe and U. The organic layer is centrifuged and its extinction is measured at 450 mμ against a blank of n-hexanol. The Mo is reduced by shaking the n-hexanol soln. with 50 ml of H_2SO_4 (14%), 1 ml of $FeSO_4$ soln. (1% in 1% aq. H_2SO_4), 10 ml of $SnCl_2$ soln. (112 g in 100 ml of conc. HCl diluted to 1 litre) and then with 5 ml of 5% NH_4SCN soln. The organic layer is centrifuged, and its extinction is measured at 460 mμ against a blank of n-hexanol. Calibration curves are prepared from standard soln. of V and Mo. The complete analysis takes ≃30 min. Over the concn. range 5 to 20 μg, each element is determined with a precision of ±1 μg at the 95% confidence level. Excess of Fe^{III}, Cu, Ni, Cr^{VI} or W^{VI} does not interfere, nor do moderate levels of Sn and Ti. J. H. WATON

5009. **Determination of plutonium in the feed and product solutions of the Windscale primary separation plant (complexometric titration).** U.K.A.E.A. (Production Group, Windscale, Cumberland). U.K.A.E.A. Report PG210(W), 1961. 12 pp.—Full experimental details are given of a procedure in which the Pu is brought into the Pu^{IV} state by treatment with HNO_2 and $(NH_4)_2SO_4.FeSO_4$ and then extracted with Hyamine 1622 into benzene. The extract is washed with HNO_3 and the benzene is evaporated; the residue is dissolved in acetone and an excess of EDTA (disodium salt) is added. The excess is then determined by titration with a standard soln. of $ZnCl_2$; dithizone is used as an indicator and the end-point is determined photometrically, the content of Pu being derived from a calibration curve. The coefficient of variation for the measurement of 100 to 500 μg of Pu is 0·33%. G. J. HUNTER

5010. **Determination of impurities in chlorine gas by gas chromatography.** J. Lacy and K. G. Woolmington (Res. Dept., African Explosives and Chemical Industries Ltd., P.O. Northrand, Transvaal, S. Africa). *Analyst*, 1961, **86**, 350–355.—With the method described, up to 1·5% (v/v) each of H, N, O and CO_2 in chlorine gas can be determined. Separation is effected by the use of two columns in series, the first containing silica gel for separating CO_2 and Cl and the second containing a molecular sieve (30 to 40-mesh Linde 5A, activated by drying at 350° for 5 hr.) to separate O, H and N. Detection is by means of a Veco thermistor-bead cell at the end of the first column and a Griffin and George thermal-conductivity cell at the end of the second column, each detector being incorporated in a separate Wheatstone bridge circuit. Argon or helium is used as carrier gas. The apparatus is described in detail. Calibration graphs of impurity content against peak height are linear and pass near the origin. For calibration it is essential to use gaseous mixtures containing Cl as the major constituent. The precision is good for H, O and N with argon as carrier gas, but, for CO_2, better results are obtained with helium. A. O. JONES

5011. **The measurement of variations in the isotopic composition of chlorine.** T. C. Hoering and P. L. Parker (Dept. of Chem., Univ. of Arkansas, Fayettesville, U.S.A.). *Geochim. et Cosmoch. Acta*, 1961, **23** (3–4), 186–199.—Chlorine is isolated as AgCl which is then converted into dry HCl for isotopic analysis by dissolving it in dil. aq. NH_3, adding excess of magnesium metal, filtering off the residual solids, evaporating the soln. to dryness and converting the solid NH_4Cl into HCl by the addition of conc. H_2SO_4 under vacuum. The HCl is frozen by means of liquid air into a trap containing P_2O_5, to remove traces of water. The isotope analyses are expressed in terms of the difference between the isotopic ratios of the sample and a standard. R. A. HOWIE

5012. **Influence of diverse ions on the determination of manganese.** W. G. Scribner and R. A. Anduze (Res. and Engng Div., Monsanto Chem. Co., Dayton 7, Ohio, U.S.A.). *Anal. Chem.*, 1961, **33** (6), 770–773.—The interference of a large number of ions with the determination of Mn by the Lingane - Karplus method (*Ind. Eng. Chem., Anal. Ed.*, 1946, **18**, 191) has been studied. Lithium, K, Rb, Cs, Be, BO_3^{3-}, Sc, Y, La, Ce^{4+}, Pr, Nd, Sm, Gd, Th, CO_3^{2-}, SiO_3^{2-}, Ti, Zr, Hf,

PO_4^{3-}, Nb, Re^{7+}, Rh, Pd, Pt, Hg, Ga, In, Tl, Ge, Sn, Pb, Bi, F^- and Br^- do not interfere. Small amounts of Sr, Ba, Ag and Ta do not interfere, but large amounts produce a ppt. containing occluded Mn; Ce^{III}, Sb^{III}, Sb^{V}, Se^{IV}, Te, Au^{III}, Ir^{IV}, Tl, Ru and I^- interfere. G. S. ROBERTS

5013. **Photometric determination of rhenium with *OO*-diethylphosphorodithioic acid.** A. I. Lazarev and V. V. Rodzaevskiĭ (Akmolinsk Agric. Inst.). *Zhur. Anal. Khim.*, 1961, **16** (2), 243–244.—*Procedure*—To 20 ml of a soln. containing 0·1 mg of Re add HCl (1:1) (5 ml) and 1% aq. *OO*-diethylphosphorodithioic acid soln. (6 ml), shake and set aside for 10 min. Add benzene (10 ml), shake for 1 min. and discard the aq. layer. Centrifuge the benzene layer and measure its extinction at 436 mμ in a 3-cm cell. The max. amounts of interfering ions that can be tolerated are—Cu 0·2 mg, As 0·6 mg, Fe^{3+} 2 mg, Sb <0·1 mg, and Mo 0·01 mg. J. W. PRICE

5014. **Solvent extraction of technetium and rhenium with pyridine or methyl-substituted pyridine derivatives from alkaline media.** S. J. Rimshaw and G. F. Malling (Isotopes Div., Oak Ridge Nat. Lab., Tenn., U.S.A.). *Anal. Chem.*, 1961, **33** (6), 751–754.—Distribution coeff. for the extraction of Tc into pyridine and ethyl methyl ketone from NaOH and Na_2CO_3 soln. of various concn. have been determined. Pyridine has been found to be the more efficient extractant, and since the uranium carbonate complex is not extracted into pyridine this system may be used for the separation of Tc and U. The presence of NO_3^- in these systems was found to decrease the affinity of Tc for pyridine, similar behaviour being found in ion-exchange resin systems. When Tc and Re are extracted together in systems containing NO_3^-, the distribution coeff. for Tc is consistently higher than that of Re. Substituted pyridines are useful for separating Tc from soln. containing appreciable amounts of NO_3^-. G. S. ROBERTS

5015. **Paper adsorption chromatography. V. Complexes of rhenium and technetium with thiourea.** T. J. Beckmann and M. Lederer (Inst. Radium, Curie Lab., Paris). *J. Chromatography*, 1961, **5** (4), 341–350 (in French).—Perrhenate and pertechnetate may be separated by chromatographing the reaction product after heating in N-HNO_3 in the presence of excess of thiourea. A separation of Tc from Mo and Mn is also described. G. RUSSELL

5016. **Colorimetric reaction for iron with diantipyrinylmethane.** L. Ya. Polyak. *Zavod. Lab.*, 1961, **27** (4), 388–389.—*Procedure for Fe in bronze*—The sample (0·2 g) is dissolved in 15 to 20 ml of HNO_3, oxides of N are boiled off, and the soln. is evaporated to a syrup with 20 ml of HCl. The soln. is diluted to 100 ml and an aliquot (5 ml) is treated with 15 ml of 0·5% diantipyrinylmethane soln., then neutralised to Congo red paper with 25% Na acetate soln. and HCl in succession, and diluted to 100 ml. The extinction is measured with a blue filter after 20 to 25 min. No interference is caused by the amounts of Mn, Ni and Al present in bronze. *Procedure for Fe in magnesium alloys*—The sample (1 g) is dissolved in 20 ml of HCl, a few drops of HNO_3 are added, and the soln. is boiled to remove oxides of N. The soln. is diluted to 50 ml and an aliquot (25 ml) is treated with 10 ml of the diantipyrinylmethane soln. and neutralised, and then diluted to 50 ml. The method is sensitive to the presence of 0·0005% of Fe. G. S. SMITH

5017. **Colorimetric determination of small amounts of nitroprusside and aquo- and ammine-pentacyanoferrates(II).** B. Jaselskis and J. C. Edwards (Dept. Chem., Univ., Ann Arbor, Mich., U.S.A.). *Anal. Chem.*, 1960, **32** (3), 381–383.—Nitroprusside is determined as the isophorone (3,5,5-trimethyl-2-cyclohexen-1-one) complex in the range $2{\cdot}0 \times 10^{-5}$M to $2{\cdot}5 \times 10^{-4}$M by measuring the extinction at 495 mμ under carefully controlled conditions, as the colour is unstable. Aquo- and ammine-pentacyanoferrates^{II} in the range 5×10^{-5}M to 5×10^{-4}M are determined as azide complexes by measuring the extinction at 560 mμ. N. E.

5018. **Colorimetric determination of small amounts of aquopentacyanoferrate(II) in the presence of ferro- and ferri-cyanide.** I. Murati and S. Ašperger (Inst. of Inorg. and Phys. Chem., Univ., Zagreb, Yugoslavia). *Anal. Chem.*, 1961, **33** (6) 809–810.—Ferricyanide interferes with the colorimetric determination of aquopentacyanoferrate $[Fe(CN)_5(H_2O)]^{3-}$ with nitrosobenzene reagent (Ašperger *et al.*, *J. Chem. Soc.*, 1960, 730) or as an azide complex (Jaselskis and Edwards, *Anal. Chem.*, 1960, **32**, 381). The interference may be eliminated by reduction of the ferricyanide to ferrocyanide with H_2O_2. G. S. ROBERTS

5019. **Determination of minor constituents in low-alloy steel by X-ray spectroscopy.** R. E. Michaelis, R. Alvarez and B. A. Kilday (Nat. Bur. of Standards, Washington, D.C., U.S.A.). *J. Res. Nat. Bur. Stand., C*, 1961, **65** (1), 71–76.—Some 20 elements can be determined satisfactorily in a curved-crystal multi-channel X-ray spectrometer by correct selection of type of crystal and detector, primary and secondary slit widths, and He or air path. The exposure time is 30 to 60 sec. (4 min. for Si). The interferences (not serious) and lower limits of detection (usually <0·01%) are listed for Ag, As, Co, Cr, Cu, Ge, Mn, Mo, Nb, Ni, Pb, S, Se, Si, Sn, Ta, Ti, V, W and Zr. The last-named interferes with the determination of Mo; B cannot be determined. In comparison with the optical emission method, the precision and accuracy are higher at concn. >1%, approx. equiv. at concn. from 0·1 to 1%, and less at concn. <0·1%. Because the X-ray method is unaffected by the chemical combination of the elements, it is preferred for the analysis of samples having differing metallurgical history. The necessary calibration curves can be constructed from the results for standard samples of iron and steel. W. J. BAKER

5020. **Photometric determination of small copper contents in iron and steel by means of biscyclohexanone oxalyldihydrazone.** S. Meyer and O. G Koch (Neukirchen Eisenwerk A.-G., Saar). *Arch. Eisenhüttenw.*, 1961, **32** (2), 67–70.—Copper can be detected by the extinction of its biscyclohexanone oxalyldihydrazone complex at 600 mμ; the colour attains full intensity immediately and is stable for 5 hr.; when citric acid is added (12·5 mg per litre for Ni contents up to 20%) the colour forms after 1·5 hr. and remains stable for a further 2 hr. Concn. of 0·5% of Ni, 1% of Co, Pb, Ti or V, 5% of Mn or W, 10% of Al or Mo and 20%

of Cr do not interfere. The Fe is separated by aq. NH_3 pptn. and Cu can be determined in the range 0·001 to 0·2%. M. LAPIDOT

5021. **Spectrophotometric determination of boron in steel and high-temperature alloys.** W. L. Karpen (Carpenter Steel Co., Reading, Pa., U.S.A.). *Anal. Chem.*, 1961, **33** (6), 738–740.—Boron is determined spectrophotometrically as the complex with tetrabromochrysazin. The reagent is used as a 10^{-3}M soln. in 96% H_2SO_4. *Procedure*—The sample (containing 2 to 25 μg of B) is dissolved by being heated under reflux with 15 ml of $HClO_4$ (60%) - H_3PO_4 (85%) (1:2) in a boron-free flask. The soln. is twice distilled (70° ±3°) with methanol (50-ml portions) into 20 ml of H_2O and 5 ml of 0·1N-NaOH in a Florence flask immersed in an ice bath. Acid-insol. material is filtered off, washed, treated as described in "ASTM Methods of Chem. Analysis of Metals", 1956, 132–136, and distilled with methanol as described above. The combined boron soln. are evaporated to dryness in a boron-free vessel under an i.r. lamp and heated for ≃20 min. When cold, the residue is dissolved in the minimum amount of 96% H_2SO_4, and added to 2·5 ml of reagent in a 25-ml flask which is stoppered and set aside for 1 hr. to develop the colour. The soln. is then diluted to 25 ml with 96% H_2SO_4 and the extinction is measured at 540 mμ against a blank. Calibration curves are prepared with H_3BO_3 soln. The method is suitable for determining B in the range 0·0002 to 0·025%. J. H. WATON

5022. **Colorimetric method for the determination of traces of cobalt.** J. Scherzer and V. Rona (C. I. Parhon University, Bucharest). *Rev. Chim., Bucharest*, 1960, **11** (12), 712–714.—*Procedure*—The test soln., containing from 0·03 to 0·13 mg of Co, is adjusted with ammonium acetate to pH 4, then treated with the reagent [a mixture of 2 ml of an ethanolic soln. of dimethylglyoxime (0·25%) and 3 ml of a freshly prepared ethanolic soln. of *o*-dianisidine (0·1%)] and diluted to 100 ml. After 30 min. the extinction is measured at 436 mμ against a water blank. The readings are then compared with a standard curve. Tervalent Fe interferes but is masked by a slight excess of a soln. of NaF (5%); Cr and Mn do not interfere, but Ni does. The error over a range of 0·3 to 1·3 μg of Co per ml is +5% to −2·4%. H. SHER

5023. **Determination of small amounts of cobalt by isotope dilution with cobalt-60.** K. F. Sporek (Bioferm Corp., Wasco, Calif., U.S.A.). *Anal. Chem.*, 1961, **33** (6), 754–758.—A highly specific method for the determination of a wide range of Co concn. in biological material, alloys, etc., is described. The method depends upon the extraction of Co from acid soln. into isobutyl methyl ketone, followed by extraction into alkaline thiocyanate soln. and spectrophotometric measurement at 620 mμ. In the determination of Co in vitamin B_{12} the coeff. of variation was 1·0%. G. S. ROBERTS

5024. **Preliminary study of the assay of elements by neutron activation, based on the formation of short-lived isotopes: quick separations and determination of losses.** D. Monnier, W. Haerdi and J. Vogel (Lab. de Chim. Minérale, Univ. Genève). *Helv. Chim. Acta*, 1961, **44** (4), 897–903 (in French).—A method for the rapid determination of Co and its separation from Fe has been developed. It comprises the toluene extraction of the complex of Co with 2-nitroso-1-naphthol. Iron, the chief element causing disturbance in assay by γ-spectrometry (^{60}Co) after activation, gives a complex with sodium fluoride - acetate - citrate and remains in the aq. phase. It has been possible to separate 0·1 μg of Co from 100 mg of Fe. The estimation of losses has been made by means of a radioactive tracer (^{58}Co). The time necessary is about 20 min. M. SULZBACHER

5025. **The use of bead reactions in quantitative micro-analysis. I. Spectrophotometric determination of cobalt and nickel in borax beads.** G. Ackermann and C. Alferi (Inst. f. anorg. Chem., Bergakad., Freiberg, Germany). *Mikrochim. Acta*, 1961, (3), 390–400 (in German).—The extinction of borax beads to which small amounts of cobalt or nickel compounds had been added was found to be proportional to the thickness of the bead and the concn. of the Co or Ni. The extinction at 580 mμ was used to determine up to 30 μg of Co to within ±1 μg. Up to 150 μg of Ni could be determined by measuring the extinction at 440 mμ; Co and Ni could also be determined together. R. M. ROWLEY

5026. **Successive separation by extraction, and determination of copper, iron and cobalt in electrolytic nickel.** S. E. Krelmer, A. V. Stogova and A. S. Lomekhov. *Zavod. Lab.*, 1961, **27** (4), 386–387.—The sample (10 g) is dissolved in aq. HNO_3 (3:2), the soln. is evaporated nearly to dryness, and the residue is dissolved in water (250 ml). An aliquot (25 ml) is diluted to 50 ml and extracted with a soln. of Pb diethyldithiocarbamate in $CHCl_3$ (5-ml portions) [concn. not stated] until the $CHCl_3$ layer is colourless. The aq. layer is washed with 5 ml of $CHCl_3$. The combined $CHCl_3$ extracts are diluted to 25 ml with $CHCl_3$ and the extinction of the Cu complex is measured with a blue filter. The aq. soln. is treated dropwise with 50% Na acetate soln. to give a pH of ≃5 and then with 30 ml of an acetate buffer soln. (pH 5·57). It is then stirred with 2 ml of ethyl acetoacetate for 1 min. and the Fe complex is extracted with $CHCl_3$ (10, 5 and 5 ml). The combined extracts are diluted to 25 ml with $CHCl_3$ and the extinction is measured with a blue filter. The aq. soln. is then treated with HCl to give a pH of ≃2, 5 ml of 40% NH_4SCN soln. and 10 ml of 2% diantipyrinylmethane soln. in 0·5N-HCl are added and the Co complex is extracted with $CHCl_3$ (10-ml portions) until the $CHCl_3$ layer is colourless. The combined extracts are treated with 15 ml of 0·2N-Na acetate and 5 ml of 0·5% nitroso-R-salt soln. The $CHCl_3$ layer is again extracted with similar portions of the same reagents and the combined aq. layers containing the Co are treated with 5 ml of aq. HNO_3 (1:1) and diluted to 200 ml after 20 min. The extinction is measured with a green filter. G. S. SMITH

5027. **Polarographic and amperometric determination of ruthenium.** N. K. Pshenitsyn and N. A. Ezerskaya. *Zhur. Anal. Khim.*, 1961, **16** (2), 196–200.—The complex $[RuCl_6]^{2-}$ is reduced to $[Ru(H_2O)Cl_5]^{2-}$ at a rotating platinum microelectrode. The diffusion current is proportional to Ru concn. over the range 1 to 200 μg per ml; the $E_{\frac{1}{2}}$ = +0·72 V *vs.* the S.C.E. Large amounts of Pt, Rh, Pd, Os, Cu, Ni, Se and Fe do not interfere.

Gold can be present in small amounts, but Ir must be absent. *Polarographic procedure*—Evaporate the chloride soln. to dryness with a little NaCl. Add 0·8M-HCl (5 ml) and 5 drops of H_2O_2 soln. and boil until colourless. Add conc. HCl (5 ml) and boil to remove Cl. Cool, dilute to 10 ml with aq. HCl (1:1) and polarograph from +0·9 to +0·5 V *vs.* the S.C.E. *Amperometric procedure (for Ru concn. up to 2 mg per ml)*—Prepare a soln. of Na_2RuCl_6 as described above and titrate with a standard soln. of quinol (1 ml ≡ 1 mg of Ru) applying a potential of +0·5 V *vs.* the S.C.E. In the presence of Au the current at the end-point is greater than zero, but the break in the titration curve is sufficiently clear in the presence of a 15-fold excess of Au. J. W. PRICE

5028. **Determination of palladium and nickel or palladium and copper when present together.** A. S. Pesis (Perm State Med. Inst.). *Zhur. Anal. Khim.*, 1961, **16** (2), 253–254.—Palladium is pptd. with 3-hydroxy-2-naphthaldehyde (**I**) at pH 2·5 to 3, and Cu and Ni by the same reagent at pH 7·2 to 7·5. *Procedure*—Adjust the pH of the soln. containing Pd and Ni to 2·5 to 3 and add an equal volume of ethanol. Add a 1% ethanolic soln. of **I** (5 ml), set aside for 25 min. and heat at 70° for 5 min. Filter and wash the ppt. with 50% ethanol, then dry it at 110° for 30 min. and weigh (factor = 0·2377). To the filtrate add a few drops of 0·25N-NaOH and solid Na acetate to adjust the pH to 7·2 to 7·5. Boil and add **I** soln. (10 ml). Set aside for 25 min., filter and wash the ppt. with 50% ethanol; dry it at 150° for 45 min. and weigh (factor = 0·1464). To determine Cu after the separation of Pd, neutralise as described above, or with aq. NH_3, and precipitate as described above (*cf.* Gusev and Kumov, *Anal. Abstr.*, 1957, **4**, 1125). J. W. PRICE

5029. **Catalytic analysis. VI. New sensitive catalytic reactions for the detection of osmium.** J. Bognár and S. Sárosi (Tech. Univ., Miskolc, Hungary). *Magyar Kém. Foly.*, 1961, **67** (5), 193–198.—The effect of OsO_4 on the kinetics of the oxidation of quinol, orcinol and 1,3-dihydroxynaphthalene by H_2O_2 was investigated; OsO_4 increased the oxidation rate in all cases, having a max. effect at pH 10·5, 10·3 and 12·4, respectively. Reagent concn. and reaction temp. both influence the oxidation rate. The three reactions were applied to the determination of trace amounts of OsO_4; 0·001 μg of OsO_4 could be detected in 5 ml of soln. The effect of the presence of other elements was investigated. It was found that EDTA (disodium salt) did not interfere with the catalytic reaction, so that this compound could be used to increase the selectivity of the method. Complex-forming compounds, *e.g.*, KCN, ammonium salts, cupferron and dimethylglyoxime, acted as catalytic poisons.

VII. Detection of sub-micro amounts of osmium by means of the potassium chlorate oxidation of 3,3′-dimethylnaphthidine. J. Bognár and S. Sárosi. *Ibid.*, 1961, **67** (5), 198–200.—The oxidation of 3,3′-dimethylnaphthidine by $KClO_3$ in the presence of Os^{8+} is completed in a few minutes, and the red compound produced can be determined colorimetrically. The application of this reaction to the detection of OsO_4 is described. The rate of reaction reaches a max. at pH 2·3 and depends on the temp. and reagent concn.; 0·001 μg of OsO_4 can be detected in 5 ml. The mechanism of the reaction and the effects of other elements are discussed. B. VASZKO

5030. **Determination of iron, calcium and silicon in calcium silicates by X-ray fluorescence.** J. W. Meyer (Johns-Manville Res. Center, Manville, N.J., U.S.A.). *Anal. Chem.*, 1961, **33** (6), 692–696.—A simple equation is given that permits the calculation of correction factors for the inert matrix effect, which is shown seriously to influence the determination of metals of low atomic wt. Thus the number of standards necessary may be reduced without decreasing the accuracy of the determination. The equation is strictly applicable only to the element of highest atomic number in the sample, but approx. values for other elements can also be obtained. The CaO is first determined, since the enhancement effect by a small Fe content is negligible; Fe_2O_3 is then obtained with the aid of corrections. If the Mg and Al contents are small, an accurate determination of SiO_2 is possible. The average errors are ≃6·9% for Fe_2O_3 (0·4 to 4%), 2·5% for SiO_2 (0 to 100%) and 2·3% for CaO (0 to 65%). J. H. WATON

5031. **Isotope dilution analysis of meteorite potassium and age determination by the K/Ar method.** D. Krummenacher (Lab. Minéral., Univ. Genève). *Helv. Chim. Acta*, 1961, **44** (4), 1054–1057 (in French).—By using this technique, the K/Ar ages of the meteorites Richardton and Murray were found to be 4·59 and 2·51 × 10^9 years, respectively. M. SULZBACHER

5032. **Determination of radioactive species produced by cosmic rays in meteorites.** M. Honda and J. R. Arnold (Univ. of California, La Jolla, Calif., U.S.A.). *Geochim. et Cosmoch. Acta*, 1961, **23** (3–4), 219–232.—A systematic fractionation scheme is described for the separation of 14 elements and the determination of cosmic-ray-produced radioactive nuclides. Particular attention is given to the determination of short-lived species not previously studied. In addition to β- and γ-counters (*cf.* Honda *et al.*, *Anal. Abstr.*, 1961, **8**, 3758), a proportional counter is used to measure the X-ray activities of isotopes of Ni, Co, Mn, Cr and V. For the Co isotopes all three counting methods are used: decay curves and absorption measurements are used to separate ^{60}Co, ^{57}Co and the pair ^{56}Co plus ^{58}Co. Absorption measurements were performed for ^{45}Ca, ^{46}Sc and ^{36}Cl, and decay or growth measurements were made for ^{44}Ti (^{44}Sc), ^{32}Si (^{32}P) and ^{41}Ca. R. A. HOWIE

5033. **Determining phosphorus in coal and coke: evaluation of volumetric, colorimetric and gravimetric methods.** F. H. Gibson and W. H. Ode (Bituminous Coal Div., Bur. of Mines, Pittsburgh, Pa.) *Rep. Invest. U.S. Bur. Min.*, 1961, No. 5743. 21 pp.—The volumetric methods investigated, *viz*, the ASTM method B for coal low in Ti, and the method of B.S. 1016:1960, the molybdenum blue colorimetric methods with $SnCl_2$ or hydrazine sulphate as reducing agent, and the colorimetric determination as molybdovanadophosphoric acid, as well as the gravimetric procedure based on the formation of a ppt. of ammonium molybdophosphate, are all about equally accurate and may be regarded as interchangeable. In all the methods the errors due to the presence of As in amounts normally found in coal are insignificant. Details of the various procedures are given. J. M. JACOBS

5034. **Spectrographic analysis of slags—the present position.** A. Argyle (B.C.I.R.A. Lab., Alvechurch, Birmingham). *B.C.I.R.A. Journal*, 1961, **9** (3),

364–376.—Work to develop a simple and accurate spectrographic method of slag analysis is reported. Results and potentialities are given of five methods: powder spark, powder arc, high-voltage a.c. arc and high-current d.c. arc, carrier distillation and X-ray emission spectrography. Fusion methods examined were pelleting and the Danielsson tape method. Examination with chemical solution techniques (*cf.* Muir and Ambrose, *Anal. Abstr.*, 1954, **1**, 2612) to improve accuracy for cupola slags showed discrepancies due to electrode treatment, and an improved procedure was developed, good standard graphs being obtained for Si, Ca, Al, Mg, Mn and Fe. Further work to secure reproducibility is outlined. C. H. COWPER-COLES

See also Abstracts—**4918**, Papers from I.U.P.A.C. Congress, Lisbon, 1956. **4922**, Determination of K, Th and Ag. **5119**, Separation of Ca and Sr. **5247**, Copper, Pb, Sn and Zn in foods. **5250**, Determination of ^{90}Sr in milk. **5279**, Determination of Mn, Cr and Fe in air. **5283**, ClO_2 in water. **5333**, Apparatus for indicating oxides in liquid metals.

3.—ORGANIC ANALYSIS

Determination of elements and radicals and of organic compounds not included in other sections. Organic industrial products, including petroleum and its products, fuels, detergents, volatile oils, cosmetics, dyestuffs, fibres, plastics, resins, paints, elastomers, leather and explosives.

5035. **Studies on the thermo-electric micro-determination of molecular weight.** C. Tomlinson (Org. Chem. Dept., Univ. Liverpool, England). *Mikrochim. Acta*, 1961, (3), 457–466 (in English).—A detailed study has been made of the conditions necessary for the accurate measurement, by means of thermistors, of the temperature difference between a drop of soln. and a drop of the solvent in a cell that is saturated with the vapour of the solvent. The temperature difference is related to the mol. wt. of the solute (*cf.* Simon and Tomlinson, *Chimia*, 1960, **14**, 301). It was found that the cell must be maintained at constant temp. (25° is suitable) to within $\pm 0{\cdot}002°$, and be shielded from radiation. Contact between the cell and the atmosphere must be minimised to reduce the effect of variations in ambient temp. To avoid convection in the cell, thermistor power dissipation must be very low, and the volume of the cell should be as small as possible. The drops of solvent and soln. must be placed on the thermistors in a reproducible way. The thermistors must be washed rapidly and thoroughly when changing the drops. The concn. of the soln. must not be too high, or a true steady state will not be reached. It was found that mol. wt. in the range 100 to 700 could be determined with a standard deviation of $\simeq 2\%$, with <10-mg samples, but an extrapolation procedure had to be used, and some determinations took >2 hr. R. M. ROWLEY

5036. **Use of induction heating in determinations of carbon and hydrogen.** D. E. Butterworth (D.S.I.R., Nat. Chem. Lab., Teddington, Middx., England). *Analyst*, 1961, **86**, 357–358.—The analysis of thermally stable compounds and polymeric compounds, particularly certain organoboron compounds, presents difficulties when the usual combustion methods are used. With the "empty tube" method of Belcher and Ingram (*Brit. Abstr. C*, 1950, 386) there was evidence that volatilisation of such samples would be complete at $\simeq 1300°$, and as this temp. could not be applied externally without risk of softening the silica tube, induction heating was applied by means of a 14-kW 350-kc/s generator in conjunction with a water-cooled copper coil placed round the tube about 1·5 in. from the main furnace. The platinum boat with the sample was placed inside a 3-in. roll of platinum foil reaching from the coil into the main furnace. The roll was wound with platinum wire to provide contact with the silica tube, so that some of the heat of the foil was transferred to the tube and condensation was prevented. Heated oxygen was passed through, the temp. of the boat was raised to 1300°, as shown by an optical pyrometer, and the combustion was completed in the normal manner. Results with standard samples, *e.g.*, acetanilide, Na tetraphenylborate and a compound containing C, H, N, B and O, were satisfactory. A. O. JONES

5037. **Quantitative organic analysis. XXVII. Kinetics and mechanism of catalysed rapid combustion in the determination of carbon and hydrogen in organic compounds.** M. Večeřa, D. Šnobl and L. Synek (Microanal. Lab., VÚOS, Pardubice-Rybitví, Czechoslovakia). *Mikrochim. Acta*, 1961, (3), 370–389 (in German).—The course of combustion in O in a tube containing Co_3O_4 as catalyst was observed by passing the exit gases through a thermal-conductivity cell. It was found that CO_2 from the combustion of sucrose and anthraquinone emerged in a single wave within 2 to 4 min., but two distinct waves were given by compounds such as triphenylbenzotristriazole, diethylene glycol, cyclohexanol and cholesteryl acetate, which leave some non-volatile residue when pyrolysed. The time for complete oxidation and purging of the combustion tube could be kept within 12 min. if the sample was covered with Co_3O_4, and determinations could be carried out on compounds such as terephthalic acid and piperidine picrate. The activity of several catalysts was studied by measuring the conversion of methane into CO_2 as a function of catalyst temperature; Co_3O_4, Cr_2O_3, NiO, MnO_2 and $AgMnO_4$ were all effective, but Co_3O_4 was superior to the others both in activity and chemical stability. The conditions necessary for very rapid combustion, based on the mechanism of combustion, were (*a*) high concn. of vapour in contact with the catalyst, (*b*) a high O concn. in the catalyst, and (*c*) a high reaction constant. These conditions could be met by volatilising the sample rapidly, having a porous catalyst with a large surface area, and maintaining the catalyst at a high temperature. Rapid purging could be attained by reducing the volume of the apparatus, reducing absorption on the catalyst by keeping it at a high enough temperature, and increasing the rate of flow of O. R. M. ROWLEY

5038. **Studies on organic micro-analysis. V. Simultaneous determination of carbon, hydrogen, halogens and sulphur.** Toshihiro Onoe, Chizuru Furukawa and Hiro Otsuka (Takamine Lab., Sankyo Co. Ltd., Shinagawa-ku, Tokyo). *Ann. Rep. Takamine Lab.*, 1959, **11**, 100–103.—The combustion method in which a quartz absorption

funnel filled with silver is used (Mitsui and Sato, *Anal. Abstr.*, 1957, **4**, 917) was improved. A side-tube was attached to the combustion tube to separate the oxygen stream into two streams, one being used for sample combustion and the other for sweeping out the combustion gas. All the gas in the tube was passed through the quartz absorption funnel and, as a result, the Mariotte bottle became unnecessary. The simultaneous determination of carbon and hydrogen is possible by connecting a carbon - hydrogen analysis tube to the end of the combustion tube. The time required for the analysis is 50 min. S. NATORI

5039. **Improvements in or relating to processes and apparatus for detecting halogen-containing compounds present in gases.** O. H. Drager. Brit. Pat. 872,671, date appl. 21.8.59; Germany, date appl. 21.8.58 and 3.8.59.—The halogen-containing gas and a combustible gas are mixed and led past heated copper which combines with the halogen. The resulting gas mixture is then led to a burner, where the flame turns greenish to indicate the presence of the halogen-containing compound. J. M. JACOBS

5040. **Semi-micro determination of fluorine and chlorine in organic compounds. IV. Determination of halogen in gases and volatile liquids.** R. D. Chambers, W. K. R. Musgrave and J. Savory (Dept. of Chem., The Univ., South Rd., Durham, England). *Analyst*, 1961, **86**, 356–357.—The method previously described (Johncock *et al.*, *Ibid.*, 1959, **84**, 245) for the determination of halogens in fluoro-, chlorofluoro- and bromofluoro-compounds was restricted to their determination in non-volatile compounds or those having fairly low b.p. A modification applicable to gases and volatile liquids having b.p. up to 80° or 90° is now described. By means of the apparatus developed, the sample is introduced into an evacuated tared glass bulb and weighed. It is then transferred to a vessel in which it is shaken with the biphenyl - sodium - dimethoxyethane complex (*loc. cit.*) and isopropyl ether to convert all the halogens into sodium halides, which are extracted with water, and the extract is analysed as previously described. The oxycyanide method is used to determine Cl, Br and I. A. O. JONES

5041. **Micro-determination of chlorine or bromine in organic compounds.** D. C. White (Res. and Devel. Dept., The Distillers Company Ltd., Great Burgh, Surrey, England). *Mikrochim. Acta*, 1961, (3), 449–456 (in English).—The titration of halides in 60% ethanol with $Hg(NO_3)_2$ soln., as described by Cheng (*Anal. Abstr.*, 1960, **7**, 3286), provides a rapid, simple and accurate finish to the determination of halogens by the oxygen-flask combustion method. The sample (containing $\simeq$1 mg of Cl or 2 mg of Br) is burned in O in a 250-ml flask containing a mixture of 0·1M-NaOH (2 ml), 100-vol. H_2O_2 soln. (2 ml) and water (1 ml). When the products of combustion have been absorbed, the stopper and platinum spiral are rinsed with water (10 ml), and the soln. is neutralised with 0·1M-HNO_3 to the yellow colour of bromophenol blue. A slight excess of 0·1M-HNO_3 (0·5 ml), with ethanol (100 ml), is added; the pH should then be 3·6. A 0·1% soln. of diphenylcarbazone in ethanol (0·5 ml) is added, and the soln. is titrated with 0·005M-$Hg(NO_3)_2$ to the first appearance of a violet colour. If the sample contains S, the SO_4^{2-} must be removed by adding $Ba(NO_3)_2$ before adjusting the pH to 3·6. If P or F is present the method of adjusting the pH must be modified. If iodine is present, H_2O_2 cannot be used in the absorption soln., and a soln. of SO_2 in water must be used. Excellent results were obtained when the method was applied to 6-mg samples of chloro- or bromo-benzoic acid, and benzylthiuronium chloride. R. M. ROWLEY

5042. **A method of general applicability for the micro-determination of chlorine, bromine and iodine.** S. Greenfield, R. A. D. Smith and I. L. Jones [Albright and Wilson (Mfg.) Ltd., Res. Dept., Oldbury, England]. *Mikrochim. Acta*, 1961, (3), 420–430 (in English).—The method described can be used with 5-mg samples of compounds containing C, N and P. The sample is burned in a slow stream of O over platinum contacts heated to 850° to 900° in a silica combustion tube, and the O and the products of combustion are drawn into an evacuated 250-ml flask containing H_2O_2 soln. When combustion is complete, the soln. in the flask is made alkaline with NaOH soln., and boiled to destroy the H_2O_2. Hydrazine sulphate is added, and the soln. is made acid to methyl orange by adding HNO_3. The halides are then titrated amperometrically with 0·01M-$AgNO_3$, with a dropping-mercury cathode. The method has been applied to the determination of Cl, Br and iodine in a variety of organic compounds; the results obtained were generally both accurate and precise. R. M. ROWLEY

5043. **Micro-determination of halogen and sulphur [by combustion] in a stream of oxygen.** E. Pella (Ist. Carlo Erba Ric. Terapeut., Milano, Italy). *Mikrochim. Acta*, 1961, (3), 472–483 (in German).—The pyrolysis of compounds containing high proportions of Cl or S, or of explosive compounds, or the volatilisation of volatile compounds, is carried out more uniformly by placing the sample in a silica tube held concentrically within the combustion tube. The pyrolysis products or the vapours are oxidised at 1000° in the combustion tube, which contains two sintered glass plates. In the determination of Cl or Br, the halogen hydracids are absorbed in 6% H_2O_2 soln. on silica beads, and titrated in ethanol with 0·005M-mercuric perchlorate, with diphenylcarbazone as indicator. In the determination of S, the oxides are absorbed in 6% H_2O_2 soln. on silica beads after passing over dry beads, and the H_2SO_4 is titrated in isopropyl alcohol with 0·005M-barium perchlorate, with thoron as indicator. A procedure for the simultaneous determination of S and Cl is given. R. M. ROWLEY

5044. **Gas-chromatographic determination of combustion products in nitrogen micro-determinations.** M. Simek and K. Tesařík (Inst. f. Anal. Chem., Univ. Brno, Czechoslovakia). *Coll. Czech. Chem. Commun.*, 1961, **26** (5), 1337–1342 (in German).—Gas chromatography is used to test for impurities in the N evolved in micro-Dumas determinations. The position of the copper spiral, the dissociation of CuO to O, and the reduction of H_2O do not affect the results. No CO appears. Optimum results are obtained by slow combustion, gradual increase in temp., and maintenance of temp. suitable for the pyrolysis of the sample (300° to 800°) and for the combustion of methane (>710°). J. P. STERN

5045. **Rapid method for micro-determination of sulphur in monomeric and polymeric organic compounds containing nitrogen, halogens and alkali metals.** A. I. Lebedeva and I. V. Novozhilova (Inst. High-Molecular-Weight Compounds, Acad. Sci. USSR, Leningrad). *Zhur. Anal. Khim.*, 1961, **16** (2), 223–225.—Combustion is carried out in a flask filled with oxygen as described by Sokolova *et al.* (*Anal. Abstr.*, 1960, **7**, 2255). The products of combustion are absorbed in 10 ml of water containing 0·5N-NaOH (1 ml) and 30% H_2O_2 soln. (3 drops) and the soln. is evaporated nearly to dryness in the presence of 0·02 mg of platinum black. Water (5 ml) and acetic acid (2:3) (5 ml) are added and the soln. is boiled for 1 min., then cooled. Ethanol (10 ml) and a 0·1% soln. of Alizarin red S (C.I. Mordant Red 3) (3 drops) are added and the soln. is titrated with 0·02N-$Ba(NO_3)_2$ to a red end-point. The sample size is from 2 to 10 mg for S contents from 2 to 40%, and the accuracy is ±0·3%. J. W. PRICE

5046. **Modifications of the Fleischer method for determining phosphorus in organic compounds.** A. M. Ryadnina (Inst. of Org. Chem., Acad. Sci., UkrSSR). *Zavod. Lab.*, 1961, **27** (4), 405–406.—Improvements in the method of Fleischer *et al.* (*Anal. Abstr.*, 1958, **5**, 2226) are described. Complete transformation of P_2O_5 to H_3PO_4 requires boiling with dil. HNO_3 for 10 to 15 min. The sample (2 to 9 mg), enclosed in filter-paper, is ignited in oxygen in a closed flask containing 10 ml of HNO_3 (1:4). The soln. is boiled for 10 to 15 min. and diluted to 100 ml. An aliquot (1 to 4 ml) is transferred to a 10-ml tube. Various amounts of a standard soln. of P are placed in similar tubes. Each soln. is diluted to 7 ml, the reagent soln. [equal vol. of 2·5% $(NH_4)_2MoO_4$ soln. and 1% basic bismuth nitrate in 7N-H_2SO_4 are mixed and treated before use with 1 g of ascorbic acid for each 100 ml] is added, and the soln. are diluted to 10 ml. After 5 min. the extinctions are measured at 650 mμ. G. S. SMITH

5047. **Studies on hydroxamic acids. XI. Micro-determination of methoxy-groups in pectins.** O. A. Guagnini, E. E. Vonesch and M. H. C. K. de Riverós (Univ. Buenos Aires, Argentina). *Mikrochim. Acta*, 1961, (3), 408–414 (in Spanish).—The pectin is hydrolysed by heating with 20% aq. NaOH in a sealed tube at 100°. An aliquot (0·3 to 0·8 ml containing 20 to 80 μg of CH_3O–) is treated with formic acid (1 ml of 80 to 90%) in a micro-distillation apparatus at 35° and the methyl formate is distilled into a receiver containing 0·1M-hydroxylamine in 70% ethanol. The formohydroxamic acid formed is determined colorimetrically, as the Fe^{3+} chelate, after treatment with $Fe(ClO_4)_3$. L. A. O'NEILL

5048. **Determination of the benzyloxy-group in benzyl esters and carbobenzyloxy-derivatives.** A. Patchornik and S. A. Ehrlich-Rogozinski (Weizmann Inst. Sci., Rehovoth, Israel). *Anal. Chem.*, 1961, **33** (6), 803–805.—The method is based on the conversion of benzyloxy-groups into benzyl bromide with 30% HBr in acetic acid. The HBr formed, after reaction of the benzyl bromide with aniline, is titrated with Na methoxide. The accuracy is within 2%. G. P. COOK

5049. **Correlation of an infra-red absorption band in the 10 to 11-micron region with the 3-substituted phenoxy-group.** H. J. Sloane (Chemical Physics Res. Lab., The Dow Chemical Co., Midland, Mich., U.S.A.). *Anal. Chem.*, 1961, **33** (6), 689–691.—The absorption band in the 10·2 to 11·3-μ region is discussed in relation to structure. In general, the band is present whether or not there are any other substituents in the phenyl ring, except in a few cases given. A study of the spectra of isomeric compounds which are not substituted in a position *meta* to the O atom reveals that there is no intense band in this region. C. B. BAINES

5050. **Determination of traces of water in hydrocarbons.** M. Kubínová, O. Vilím and V. Svoboda (Res. Inst. for Synth. Rubber, Gottwaldov, Czechoslovakia). *Coll. Czech. Chem. Commun.*, 1961, **26** (5), 1320–1324 (in German).—The turbidity formed by reaction of $TiCl_4$ (introduced as vapour) with the H_2O in the sample is determined nephelometrically; 10^{-4} % of H_2O can thus be determined by means of a calibration curve. The method is rapid and requires no special equipment. J. P. STERN

5051. **Determination of trace quantities of acetylene in ethylene by gas chromatography.** R. A. L. Paylor and R. Feinland (Central Res. Div., American Cyanamid Co., Stamford, Conn., U.S.A.). *Anal. Chem.*, 1961, **33** (6), 808–809.—This method permits the direct injection of the sample, thus saving time and labour. A flame-ionisation detector is used and the column (5·5 ft. × 0·25 in.) of 20% hexamethylphosphoramide on 60 to 80-mesh Chromosorb is operated at 27° ± 1·5°. Under these conditions the limit of detection for acetylene is 0·03 p.p.m. and the analysis is completed in about 12 min. C. B. BAINES

5052. **Polarographic determination of chloroform and dichloromethane when present together in hydrochloric acid.** M. I. Levinskii, M. M. Filimonova and Zh. D. Gudzenko. *Zavod. Lab.*, 1961, **27** (5), 546–548.—Derivative polarographic waves for $CHCl_3$ ($E_{\frac{1}{2}}$ = −1·55 V) and dichloromethane ($E_{\frac{1}{2}}$ = −2·35 V) are obtained in a basal soln. of 0·05M-tetraethylammonium hydroxide in 75% dioxan. G. S. SMITH

5053. **Determination of ethylene and propene oxides when present together by a polarographic method.** N. N. Kosenko (Kemerovo Nitrate Fertiliser Works). *Zavod. Lab.*, 1961, **27** (5), 542–544.—The sample containing 0·08 to 0·09 g of the oxides is measured into ice-cold 10% H_2SO_4 soln. (50 ml) and the soln. is diluted to 100 ml at room temp. An aliquot (10 ml) is heated with 10 ml of water and 8 ml of 0·35N-HIO_3 to form aldehydes. The soln. is distilled, and the distillate is collected in 50 ml of water, which is then diluted to 100 ml. An aliquot (9 ml) is mixed with 1 ml of 0·1N-LiOH and the waves of formaldehyde and acetaldehyde are measured. Standardisation is by the method of additions. G. S. SMITH

5054. **Detection of organic peroxides.** G. H. Foxley (Sterling Moulding Materials Ltd., Dale St., Stalybridge, Ches., England). *Analyst*, 1961, **86**, 348–349.—Three tests are described. (*i*)—To a drop of a soln. of the peroxide in benzene allowed to dry on filter-paper is added a drop of a soln. of KI (15 g in 10 ml of air-free water, 100 ml of air-free methanol and 1 ml of glacial acetic acid). In the presence of hydroperoxides or aryl or ketone peroxides a stain of iodine forms and the test may be sensitised with starch. A soln. of titanic sulphate

in 10% H_2SO_4 (prep. described) gives an orange colour with hydroperoxides and ketone peroxides only. (*ii*)—To one drop of tetramethyl base soln. [5% (w/v) of 4,4′-tetramethyldiaminodiphenylmethane in benzene] dried on filter-paper is added a drop of a soln. of the peroxide in benzene. A blue colour forms with acyl peroxides and peresters. (*iii*)—Test (*ii*) is made on filter-paper previously soaked in a soln. of $(NH_4)_2SO_4.FeSO_4$ or $(NH_4)_2SO_4.Fe_2(SO_4)_3$ and dried. Alternatively, the iron salt soln. may be applied to the colourless spot left as a negative result with test (*ii*). Test (*ii*) is thus extended to include hydroperoxides, ketone peroxides and, when the ferric salt is used, some dialkyl peroxides. The limits of detection with 22 organic peroxides are given. Hydrogen peroxide reacts in tests (*i*) and (*iii*) only.

A. O. JONES

5055. **Rapid spectrophotometric determination of microgram amounts of lauroyl and benzoyl peroxide.** P. R. Dugan (Syracuse Univ. Res. Corp., N.Y., U.S.A.). *Anal. Chem.*, 1961, **33** (6), 696–698.—Lauroyl and benzoyl peroxides catalyse the colorimetric reaction between *p*-amino-*NN*-dimethylaniline sulphate and methanol, the intensity of the colour being a function of the peroxide concn. under controlled conditions. The extinction is measured at 560 mμ. The precision (95% confidence limits) is ±4·1% (8 determinations of each peroxide).

G. P. COOK

5056. **Analysis of the products of manufacture of isobutyl alcohol and n-butanol by gas-liquid chromatography.** E. T. Sarancha and A. P. Dubovikova (Lisichansk Chem. Combine). *Zavod. Lab.*, 1961, **27** (4), 398–399.—The method is based on the use of dinonyl phthalate on diatomite. The contents of propanol, butanol, and isobutyl and isoamyl alcohols are determined.

G. S. SMITH

5057. **Dichromate oxidation of glycols.** C. L. Whitman, G. W. Roecker and C. F. McNerney (U.S. Naval Propellant Plant, Indian Head, Md., U.S.A.). *Anal. Chem.*, 1961, **33** (6), 781–782.—By control of acidity and reaction time, the oxidation by $Cr_2O_7^{2-}$ can be stopped after the consumption of 16 and 20·5 equiv. of $Cr_2O_7^{2-}$ per mole of diethylene glycol and triethylene glycol, respectively. This forms the basis of a method in which recoveries of 99·5% are obtained for concn. of 1% or less of either of these glycols in aq. soln.

G. P. COOK

5058. **Specific sensitive assay of glycerol and L-α-glycerophosphate.** J. J. Boltralik and H. Noll (Dept. of Microbiol., Sch. of Med., Univ. of Pittsburgh, Pa., U.S.A.). *Anal. Biochem.*, 1960, **1** (3), 269–272.—The two-stage enzymic assay of glycerol described by Bublitz and Kennedy (*J. Biol. Chem.*, 1954, **211**, 951) has been modified so that glycerokinase and glycerophosphate dehydrogenase can be used in the presence of each other with adenosine triphosphate and DPN^+. The product, DPNH, is determined spectrophotometrically in 50% aq. ethanol at 340 mμ. Recoveries of 0·2 to 1 μmole of glycerol were 96·5 ±2%.

J. R. HUDSON

5059. **A method for the determination of reducing disaccharides in the presence of aldohexoses.** B. B. Jørgensen and O. B. Jørgensen (Tech. Univ., 14 Odensegade, Copenhagen, Denmark). *Acta Chem. Scand.*, 1961, **15** (3), 710–711 (in English).—This method requires <0·180 mg of disaccharide and can be used to determine glucosidases that are difficult to determine with the Tauber and Kleiner reagent (*J. Biol. Chem.*, 1932, **99**, 249). The sugar mixture is oxidised at room temp. with alkaline hypobromite solution to the corresponding aldonic and aldobionic acids. Anthrone gives a blue-green colour only with aldobionic acid when heated in H_2SO_4 so that, after adding urea to destroy the excess of hypobromite, the determination is completed by an anthrone method such as that of McCready (*Anal. Chem.*, 1950, **22**, 1156). Two blanks (containing 10^{-6} mole of monosaccharide) and two standards (containing $0·5 \times 10^{-6}$ mole of disaccharide) are included in each set of samples. Small amounts of protein and acetate or citrate buffer do not interfere, but the pH of the sample and of the hypobromite solution should be ≃10 to avoid over-oxidation of the aldobionic acid. Relative errors of single determinations vary between +2% and −3% for 0·05 to $0·5 \times 10^{-6}$ mole of disaccharide.

J. HALL

5060. **New spectrophotometric method for the determination of acraldehyde in combustion gases and in the atmosphere.** I. R. Cohen and A. P. Altshuller (R. A. Taft San. Engng Center, Cincinnati, Ohio, U.S.A.). *Anal. Chem.*, 1961, **33** (6), 726–733.—The method is based on the reaction of acraldehyde with 4-hexylresorcinol in ethanol-trichloroacetic acid in the presence of $HgCl_2$ to produce a blue colour, which is measured at 605 mμ. Other aldehydes and ketones do not interfere. The accuracy is within ±5%.

G. P. COOK

5061. **Determination of acetone and diacetone alcohol in admixture by ultra-violet absorption.** A. Basiński and A. Narebska (Copernicus Univ., Toruń, Poland). *Roczn. Chem.*, 1961, **35** (4), 1131–1141.—The absorption spectra of acetone and diacetone alcohol (4-hydroxy-4-methylpentan-2-one) have been determined in ethanol. A method for their determination in mixtures, based on measurements of the extinctions at 253·0 mμ and 285·0 mμ, is described.

N. E.

5062. **Micro-scale separation of mixtures of acids using ion-exchange resins.** G. J. Lawson and J. W. Purdie (Coal Constitution Lab., Dept. of Mining and Mineral Engng, The Univ., Edgbaston, Birmingham, England). *Mikrochim. Acta*, 1961, (3), 415–419 (in English).—The mixture of aliphatic carboxylic acids is first resolved into a number of fractions by gradient elution chromatography, with Dowex-1 resin as stationary phase and 0 to 90% formic acid soln. as mobile phase. The fractions are then spotted on Whatman No. 1 paper and developed by the ascending technique with ethyl acetate-acetic acid-water (2:1:1) containing 0·05% of Na acetate and 0·015% of bromophenol blue. The column (1·5 mm × 100 mm) of anion-exchange resin is connected by a capillary siphon to a reservoir, containing water (1·1 ml), which is connected by capillary tubing to a second reservoir containing 90% formic acid soln. (1 ml). Enough of each fraction is collected to form a spot 1 cm in diam. on a line 3 cm from the edge of the filter-paper, dried in a stream of warm air, and re-spotted twice more. The method has been applied to the separation of 200 μg each of 12 acids in 0·2 ml of water. The acids were characterised by the order in which they were eluted from the ion-exchange column, and by their R_F values on the paper chromatogram.

R. M. ROWLEY

5063. **Potentiometric titration of multicomponent acid mixtures in non-aqueous solutions.** A. P. Kreshkov, L. N. Bykova and N. A. Kazaryan (D. I. Mendeleev Chem. and Technol. Inst., Moscow). *Zhur. Anal. Khim.*, 1961, **16** (2), 129–134.—The use of tetraethylammonium hydroxide for the titration of mixtures of acids (*Anal. Abstr.*, 1960, **7**, 2775) is extended to systems containing up to 5 components, and further titration curves are given. Mixtures of phenol derivatives and acetic and benzoic acids with organic and inorganic acids can be determined with good accuracy.
J. W. PRICE

5064. **The effect of pH on the R_F values of organic acids in paper chromatography with neutral solvents.** A. Panek and R. Leibsohn (Microbiol. Lab., Nat. Coll. Chem., Rio de Janeiro, Brazil). *J. Chromatography*, 1961, **5** (4), 308–314 (in English).—The acids were used in 0·1 or 1% soln. either in water or in phosphate buffer (pH 8). Ascending chromatograms were run on Macherey-Nagel No. 261 paper for 20 hr. For dicarboxylic, hydroxy- and keto-acids, n-propanol - H_2O (7:3) was the solvent; for amino-acids, 77% ethanol was used. Conventional spray reagents were used for detection. It was found that the R_F of an acid, the soln. of which was adjusted to pH 1 before chromatography, was constant irrespective of the acid used (HCl, H_2SO_4 or trichloroacetic acid). For a given pH, the R_F was constant irrespective of whether the soln., before pH adjustment, was in water or buffer. With the amino-acids, the R_F of glutamic and aspartic acids increased greatly ($\simeq$100%) as the pH of the original soln. was reduced from 5 to 1; their amides showed a smaller effect. Other amino-acids were unaffected. Oxalic and malonic acids increased in R_F when the pH of their soln. was reduced; on the other hand, succinic and adipic acids were unaffected. Hydroxy- and keto-acids showed very large effects. The results are interpreted in terms of the effect of substituents on the dissociation constant of the —COOH group. G. RUSSELL

5065. **The separation of C_6 to C_{12} dibasic acids in the presence of monobasic acids. A simple procedure by paper chromatography.** J. L. Occolowitz (Defence Standards Lab., Maribyrnong, Victoria, Australia). *J. Chromatography*, 1961, **5** (4), 373–374 (in English).—The silica gel method of Zbinovsky (*Anal. Abstr.*, 1955, **2**, 2756) has been modified for use on paper. Shake a mixture of 2-methoxyethanol (9 parts) and H_2O (1 part) with an equal vol. of n-butyl ether. Pass a sheet of Whatman No. 1 paper (57 cm × 46 cm) through the lower layer in a shallow dish and allow to drain for 5 min. at room temp. Dissolve the acid mixture in acetone and apply 0·02-ml aliquots ($\equiv$ 10 to 50 μg of acid) 8 cm from one end. Develop by the descending method with the upper layer of the solvent mixture in a tank containing dishes of both phases. After 18 to 24 hr., remove the paper and dry for 5 min. at 105°. Spray with bromocresol green soln. (0·1% in 0·05% aq. $NaHCO_3$). R_F values are quoted. Monobasic acids move near the solvent front. G. RUSSELL

5066. **General method for the determination of anhydrides and chloroanhydrides of carboxylic acids.** L. M. Litvinenko, D. M. Aleksandrova and V. G. Napadallo (A. M. Gorky Karkov State Univ.). *Zhur. Anal. Khim.*, 1961, **16** (2), 226–228.—*Procedure*—Add 0·04 millimole of the sample to a mixture of 0·2M-*m*-chloroaniline in benzene or nitrobenzene (0·6 ml) and glacial acetic acid (0·4 ml) and heat to 65° to 70° on a water bath, in a flask equipped with an air-condenser, for up to 1 hr. Transfer to a beaker, rinsing the reaction flask with a total of 10 to 15 ml of acetic acid, add 8% HCl (1 ml) and KBr (0·5 g) and dilute to 40 to 50 ml. Titrate the excess of amine potentiometrically with 0·01M-$NaNO_2$ by the method of Litvinenko and Grekov (*Anal. Abstr.*, 1956, **3**, 1069).
J. W. PRICE

5067. **Determination of small amounts of amine in aqueous solution.** P. J. Lloyd and A. D. Carr (Chem. Dept., Univ. of Cape Town, Rondebosch, S. Africa). *Analyst*, 1961, **86**, 335–338.—To the sample soln., the pH of which is adjusted with H_3PO_4 to 1·4, are added $Co(NO_3)_2$ and NaSCN to give a concn. of 40 g of each salt per litre, the pH being then re-adjusted to 1·4. The blue complex is extracted with a 20% (v/v) soln. of amyl alcohol in kerosine. After 10 min. the separated organic layer is filtered, and the extinction of the filtrate is measured at 625 mμ against a reagent blank and referred to a calibration graph. Beer's law applies over the range of concn. investigated (5 to 200 p.p.m.), and the method is accurate to within $\pm$1% for concn. down to 5 p.p.m. of amine in the aq. soln. Iron interferes. For small amounts (<0·5 g of Fe^{3+} per litre), slightly more Na_2HPO_4 than is required to discharge the colour of $Fe(SCN)_3$ is added before the extraction of the amine complex. With larger amounts, the Fe^{3+} are reduced with ascorbic acid, 10 ml of a soln. containing 20% each of $Co(NO_3)_2$ and NaSCN is added and at least 10 g of Na_2HPO_4, and the pH is adjusted to 2·5 with H_3PO_4 before the extraction. The method was developed for the determination of amine loss in the aq. layer when uranium is extracted from aq. liquids with long-chain amines.
A. O. JONES

5068. **Mercurimetric micro-determination of hydrochlorides, hydrobromides, and methyl iodide compounds of organic bases at the tenth-milligram level.** A. Dirscherl and F. Erne (Chem. Forschungsabt., F. Hoffmann-La Roche & Co. A.-G., Basel, Switzerland). *Mikrochim. Acta*, 1961, (3), 401–407 (in German).—A procedure is given for the titration of the halides of quaternary nitrogen bases. The sample (0·3 to 1·5 mg) is weighed in a small platinum boat on an ultra-micro balance, and dissolved in a mixture of water (1 ml) and isopropyl alcohol (4 ml). Diphenylcarbazone (3 drops of a 0·1% soln. in isopropyl alcohol) and 0·1M-$HClO_4$ (1 drop) are added, and the soln. is titrated with 0·005M-mercuric perchlorate to the first appearance of a violet colour. A series of 11 determinations of iodine in *N*-methylisoquinolinium iodide gave a mean value of 46·83% (theoretical 46·81%) with a standard deviation of 0·18%. The halogen contents of the hydrohalides of 14 other bases differed from the theoretical values by not more than 0·24% (absolute). R. M. ROWLEY

5069. **Determination of mono-, di- and trichloroamine by ultra-violet absorption spectrometry.** F. W. Czech, R. J. Fuchs and H. F. Antczak (Food Machinery and Chem. Corp., Carteret, N. J., U.S.A.). *Anal. Chem.*, 1961, **33** (6), 705–707.—The method is based on the different u.v. absorption characteristics of the chloroamines and chlorine in anhydrous CCl_4. Recoveries ranged from 95 to 108%. G. P. COOK

5070. **Determination of thiols by protometry in an organic solvent.** F. Pellerin and J.-A. Gautier (Fac. de Pharm., Paris, France). *Ann. Pharm. Franç.*, 1961, **19** (2), 81–85.—The H^+ liberated in pyridine soln. when a silver salt is added to a compound containing a mercapto-group are titrated with ethanolic KOH. *Procedure*—The weighed sample (1 milli-equiv.) is dissolved in 15 ml of pyridine (or if necessary in 5 ml of water, ethanol or acetone, with subsequent addition of 10 ml of pyridine), then 0·5M-$AgNO_3$ in pyridine (5 ml) is added and after 15 min. the soln. is titrated with 0·1N-KOH in ethanol, potentiometrically or with 0·1% ethanolic thymol blue or 1% ethanolic thymolphthalein as indicator. A reagent blank must be determined. E. J. H. BIRCH

5071. **Mass spectra of aliphatic thiols and sulphides.** E. J. Levy and W. A. Stahl (Atlantic Refining Co., Philadelphia, Pa., U.S.A.). *Anal. Chem.*, 1961, **33** (6), 707–721.—To aid in the identification of thiols and sulphides for which calibration compounds are not available, mass-spectral structure correlations were developed which enabled a compound to be identified as either a thiol or a sulphide and then assigned to a structural class. The mass spectra of 29 thiols and 31 sulphides were examined. G. P. COOK

5072. **Analysis of metallo-organic compounds. Dithizonates of di- and tri-alkyl-lead chlorides.** R. Barbieri, G. Tagliavini and U. Belluco (Centro Stud. Chim. Nucl., Univ. Padova, Italy). *Ric. Sci.*, 1960, **30** (12), 1963–1968.—Mixtures of di- and tri-alkyl-lead chlorides in aq. solution are analysed by conversion into dithizonates, which are extracted with $CHCl_3$ and determined spectrophotometrically. The absorption maxima are at 498 mμ and 430 mμ for R_2PbCl_2 and R_3PbCl (R = CH_3 or C_2H_5), respectively, and the proportions are obtained by differential analysis. If Pb^{2+} are present they are complexed with EDTA to prevent interference. L. A. O'NEILL

5073. **Infra-red and Raman spectra of some tin alkyls.** C. R. Dillard and J. R. Lawson (Tennessee A. and I. State Univ., Nashville, Tenn., U.S.A.). *J. Opt. Soc. Amer.*, 1960, **50** (12), 1271–1274.—Spectra, in the wavelength region 2 to 25 μ, of tetramethyl-, ethyltrimethyl-, diethyldimethyl-, triethylmethyl- and tetraethyl-tin have been obtained as well as Raman spectra for tetramethyl- and tetraethyl-tin. N. E.

5074. **Analysis of organotin compounds.** K. Bürger (Farbwerke Hoechst A.-G., Gendorf Obb, Germany). *Z. LebensmittUntersuch.*, 1961, **114** (1), 1–10.—Salts of the di- and tri-basic di- and mono-alkyl (or aryl) compounds of tin can be detected by the deep-blue complex formed with catechol violet (**I**); the tetra-alkyl (or aryl) compounds and the salts of the monobasic compounds do not give the reaction. The blue complexes are quant. destroyed by EDTA; diphenyltin diacetate can be accurately determined in the presence of triphenyltin acetate by adding a soln. of **I** to the test soln. in methanol and titrating to a yellow colour with EDTA soln. (disodium salt). The phenyl groups in these compounds (non-basic to tribasic) can be determined by reduction to benzene with zinc and HCl at 60° and nitration of the liberated benzene to *m*-dinitrobenzene, which is then coupled in alkaline soln. with ethyl methyl ketone to form a coloured compound which is determined spectrophotometrically at 565 mμ. The accuracy is within 10 to 20%. The pptd. blue complexes can also be analysed by this method, as the complexed **I** is not reduced to benzene. Residues of the phenyltin compounds are extracted from dried powdered plant material (1 to 2 g) by heating under reflux with $CHCl_3$ (2 × 60 ml) for 20 min.; an aliquot of the filtered extract is evaporated to dryness in the reaction flask before the determination of phenyl radicals. Milk (5 litres) is first spray-dried; the powder is heated with $CHCl_3$ (2000, 1000 and 500 ml for 45 min. each). The pasty residue obtained after evaporation of the filtered extract is mixed with an absorbent material (preferably Sterchamol); half of the material, well mixed with zinc dust (20 to 30 g), can be used for the determination of phenyl radicals, and half (without zinc) for the determination of Sn by the dithiol method. Milk gives an average blank value equiv. to 5 μg of triphenyltin acetate per litre, with a standard deviation of 6 μg; recoveries of this substance added to milk are satisfactory; a positive result cannot be claimed unless the amount found is >11 μg per litre. P. S. ARUP

5075. **Chromatography of organic compounds. IX. Separation of organotin compounds by paper and gas chromatography.** J. Franc, M. Wurst and V. Moudrý (Res. Inst. for Org. Synth., Pardubice-Rybitví, Czechoslovakia). *Coll. Czech. Chem. Commun.*, 1961, **26** (5), 1313–1319 (in German).—A series of alkyltin chlorides, prepared from $SnCl_4$ by the Grignard reaction, has been studied and their physical properties, including R_F and R_M values, are listed. For paper chromatography (Whatman No. 2 paper) a 30% soln. of petroleum (boiling-range 190° to 275°) in benzene is used as the stationary phase and ethanol - acetic acid - water (20 : 1 : 14) as the mobile phase. For gas chromatography, Chromosorb and Celite columns are impregnated with silicones (20 to 30%), N is used as carrier and a thermal-conductivity cell as detector. Separation by gas chromatography is not very successful. In paper chromatography, in which the spots are revealed with dithizone after 16 to 18 hr. at 20°, the same sequence and differences of R_M values are obtained in the different alkyl series (ethyl, propyl, isopropyl, butyl, isobutyl and octyl) for different degrees of substitution (mono-, di-, tri- and tetra-alkyl). Thus the behaviour of the derivatives on the paper is predictable by means of rectilinear plots. J. P. STERN

5076. **Chromatographic analysis of the products of manufacture of phenol from isopropylbenzene.** A. N. Korol' (Groznenski Chem. Works). *Zavod. Lab.*, 1961, **27** (4), 396–398.—The method is based on the use of a column of diatomite containing tritolyl phosphate, with H as carrier gas. An internal standard, *p*-di-isopropylbenzene, is used to give quant. results for isopropylbenzene, α-methylstyrene, acetophenone and phenol. G. S. SMITH

5077. **Analysis of mixtures of some monohydric phenols by partition chromatography and ultraviolet spectrophotometry.** I. M. Belova (All-Union Sci. Res. Inst. Metallurg. Thermal Engng, Sverdlovsk). *Zhur. Anal. Khim.*, 1961, **16** (2), 229–236.—Mixtures of phenol and the isomeric cresols were separated by the method of Sweeney and Bultman (*Anal. Abstr.*, 1954, **1**, 108) with water

on silicic acid as the stationary phase and 2,2,4-trimethylpentane as the mobile phase. The resulting eluates were examined spectrophotometrically. The first 100 ml consisted of pure solvent, and the next 65 to 100 ml contained *o*-cresol, having an absorption max. at 271·8 mμ. The next 150 to 175 ml contained a mixture of *m*- and *p*-cresols. These were determined by measurements of extinctions at 273 mμ and 285·75 mμ, and calcuation from the known absorption coefficients of the two components. The final 200 ml of eluate contained the phenol, having an absorption max. at 270·8 mμ. Higher homologues interfere in the determination of *o*-cresol. The compounds named were determined in industrial effluents by this procedure, after preliminary separation of the phenolic fraction.

J. W. PRICE

5078. **Ultra-violet fluorescence of sulphosalicylic acid for the detection and differentiation of ketoses and aldoses.** E. S. Rorem (Western Regional Res. Lab., U.S. Dept. Agric., Albany, Calif.). *Anal. Biochem.*, 1960, **1** (3), 218–220.—Developed and dried chromatograms of ketoses and aldoses are dipped in a 1% soln. of sulphosalicylic acid in acetone, dried in air and viewed under u.v. light. The spots are marked and the chromatogram is heated at 95° for 5 min. Spots due to ketoses show a tan colour, while the other spots are unchanged. When viewed under u.v. light of 2537 A, the ketose spots appear dark, and under radiation of 3600 A they show an intense golden fluorescence; ≃5 μg can be detected.

J. R. HUDSON

5079. **Bromatometric determination of cyclohexanone oxime.** O. M. Podurovskaya, R. A. Kutilina and N. I. Efimova. *Zavod. Lab.*, 1961, **27** (4), 403–405.—The sample (1 g) is dissolved in 100 ml of acetic acid. To 5 ml of the soln. mixed with 25 ml of 0·25N-$KBrO_3$ is added 20 ml of an aq. soln. containing 14% of H_2SO_4 and 34% of H_3PO_4, and the containing flask, fitted with a tap-funnel, is kept closed for 5 min. Then 15 ml of 10% KI soln., followed by 50 ml of water, is added and the liberated iodine is titrated with 0·1N-$Na_2S_2O_3$.

G. S. SMITH

5080. **Chromatographic separation of substituted carboxylic acids, phenols, and aromatic carbon-ring and heterocyclic compounds, on modified cellulose papers.** F. Micheel and W. Leifels (Org.-chem. Inst., Univ. Münster, Germany). *Mikrochim. Acta*, 1961, (3), 467–471 (in German).—An improved method for the preparation of polyethyleneimine-treated cellulose paper is described. The paper has been used for the separation of the following groups of compounds—dihydroxybenzoic acids, benzenetricarboxylic acids, pyridinecarboxylic and pyridinedicarboxylic acids, aliphatic α,ω-dicarboxylic acids, aliphatic α-keto-acids, aminobenzoic acids, fluorobenzoic acids, dinitrophenols, hydroxybenzaldehydes, chloronitrobenzoic acids and aminonitrophenols. For the separation of polynuclear aromatic hydrocarbons, benzoylcellulose paper can be used. Acetylcellulose paper is useful for the separation of hydrophobic heterocyclic bases.

R. M. ROWLEY

5081. **Polarographic determination of terephthalic acid.** V. D. Bezuglyĭ and E. Yu. Novik (Kharkov Dental Materials Works). *Zavod. Lab.*, 1961, **27** (5), 544–545.—The sample is neutralised to phenolphthalein with 0·3N to 0·5N-tetraethylammonium hydroxide and the soln. is diluted to 50 ml. An aliquot (0·5 ml) is mixed with 9 ml of 0·5N-$MgCl_2$ or $CaCl_2$, O is removed and a polarogram over the range −1 to −2·2 V is obtained. A second polarogram is obtained after addition of 0·5 ml of 0·05N-terephthalic acid for calculating the content in the sample.

G. S. SMITH

5082. **Identification and separation of secondary aliphatic amines as *NN*-dialkyl-3,5-dinitrobenzamides.** B. Volakova (Chem.-Tech. Inst., Pardubice, Czechoslovakia). *Coll. Czech. Chem. Commun.*, 1961, **26** (5), 1332–1336 (in Russian).—3,5-Dinitrobenzoyl chloride is used for the identification of primary and secondary amines as their 3,5-dinitrobenzamides. The physical constants of 8 derivatives are tabulated. The dinitrobenzamides are separated chromatographically on paper impregnated with a 30% soln. of formamide in ethanol. The chromatograms are moistened with a mixture of Na ethoxide and acetone (1:1). The intense violet colour developed is unstable and after several minutes fades and turns brown. Identification is by means of the R_F values (tabulated).

A. BURWOOD-SMITH

5083. **Spot-test detection and colorimetric determination of aromatic amines and imino heteroaromatic compounds with 3-methylbenzothiazolin-2-one hydrazone.** E. Sawicki, T. W. Stanley, T. R. Hauser, W. Elbert and J. L. Noe (R. A. Taft San. Engng Center, Cincinnati, Ohio, U.S.A.). *Anal. Chem.*, 1961, **33** (6), 722–725.—Limits of identification, wavelength maxima and molecular extinction values are given for about 100 compounds with this reagent. Many carcinogenic amines can be determined by this colorimetric method. Aliphatic aldehydes, ketones and phenols seriously interfere.

G. P. COOK

5084. **New method for the spectrophotometric determination and characterisation of *NN*-dialkylanilines, diphenylamines and carbazoles using 5-nitroisatin.** E. Sawicki, T. W. Stanley and W. Elbert (Robert Taft San. Engng Center, U.S. Dept. of Health, Education and Welfare, Cincinnati 26, Ohio). *Mikrochim. Acta*, 1961, (3), 505–511 (in English).—The 5-nitroisatin chloride reagent is prepared by adding PCl_5 (2·1 g) to 5-nitroisatin (1·2 g) dissolved in hot *o*-dichlorobenzene (70 ml) and boiling the mixture for 3 min. The soln. is cooled and made up to 100 ml. *Procedure*—Add reagent (5 ml) to a soln. of the amine in *o*-dichlorobenzene (5 ml), and pass dry HCl through the mixture for 1 min. Heat the soln. for 5 min. on a boiling-water bath, then cool it, and dilute to 50 ml. Measure the extinction of the soln. (against a blank) in the range 400 to 800 mμ. Dialkylanilines give characteristic absorption spectra; the strongest band lies at 665 to 675 mμ; the extinction coefficient of this band varies from 11,000 (for *NN*-diethylaniline) to 37,000 (for *N*-benzyl-*N*-methylaniline). *N*-Alkylcarbazoles also give strong absorption in this region, but the shapes of the spectra are quite different. *N*-Methyldiphenylamine gives a spectrum similar to those of dialkylanilines. Aniline, *N*-methylaniline, phenol, 1-naphthol and indole react with the reagent, but the extinction coefficients of the products are less than 3000 at 665 mμ.

R. M. ROWLEY

5085. **Determination of small contents of the components in a binary mixture of β- and γ-picoline.** J. Souček and J. Vřešťál (Military Acad. "A. Zápotocký", Brno, Czechoslovakia). *Coll. Czech. Chem. Commun.*, 1961, **26** (5), 1466–1469 (in German).—The region 400 to 700 cm^{-1} is preferable to the 750 to 800-cm^{-1} region for the i.r. analysis of mixtures of β- (**I**) and γ-picoline (**II**), particularly for the control of purity. The bands used are at 628 cm^{-1} for **I** and at 484 cm^{-1} for **II** and do not overlap. Calibration curves, prepared by step-wise addition of the impurity to its isomer, are linear up to a content of 8% of **I** in **II**, and 4% of **II** in **I**. The accuracy is ±0·2% absolute. For higher concn., soln. in cyclohexane may be used. Mixtures of pyridine (band at 405 cm^{-1}) and α-picoline (473 cm^{-1}) may be analysed similarly. J. P. Stern

5086. **Estimation of 4,7-dichloroquinoline.** Arun Bose, A. Ray Chaudhuri and T. N. Ghosh (Bengal Immunity Res. Inst., Calcutta). *J. Instn Chem., India*, 1961, **33** (1), 16–18.—The method described is based on the reaction of 4,7-dichloroquinoline with Na methoxide in anhydrous methanol to produce 7-chloro-4-methoxyquinoline and NaCl, which is determined volumetrically or gravimetrically, with $AgNO_3$. The addition of nitrobenzene before the titration of excess of $AgNO_3$ with NH_4SCN is helpful in masking the colour of the crude sample. I. E. Jones

5087. **A new volumetric method for the determination of piperazine.** I. Grecu (Fac. Pharm., Univ., Cluj, Romania). *Rev. Chim., Bucharest*, 1960, **11** (12), 714–715.—The method is based on the titration of the excess of NH_4SCN after pptn. of the complex $[Cd(C_4H_{10}N_2)](SCN)_2$. *Procedure*—An aq. soln. is treated with a known excess of 0·1N-NH_4SCN (50 to 70 ml) and an excess of 10% Cd acetate or sulphate soln. (5 to 10 ml). The mixture is diluted to 100 ml and well shaken, and the ppt. is allowed to settle for 10 to 15 min. It is then filtered off, the first 10 ml of filtrate being rejected. The remainder of the filtrate is titrated with 0·1N-$AgNO_3$. H. Sher

5088. **Determination of impurities in hydrocarbons by means of the absorption of tritium bremsstrahlung. Preliminary communication.** P. F. Csányi, G. Horányi and O. Kaposi (L. Eötvös Sci. Univ., Budapest). *Magyar Kém. Foly.*, 1961, **67** (5), 232.—Sulphur and lead, which are present as impurities in hydrocarbon mixtures, exhibit marked absorption of tritium bremsstrahlung, the extent of which may be used for their determination in such mixtures. The method described, which is similar to that of Kannuna (*Anal. Abstr.*, 1958, **5**, 1285), can be used to determine S with an accuracy of ±0·015% (absolute). B. Vaszko

5089. **Identification of hydrocarbon peaks in gas chromatography by sequential application of class reactions.** R. Rowan, jun. (Anal. Res. Div., Esso Res. and Engng Co., P.O. Box 121, Linden, N.J., U.S.A.). *Anal. Chem.*, 1961, **33** (6), 658–665.—By proper application of class reactions, conducted in a closed system which is an integral part of the apparatus, peaks from a complex mixture are identified as n-olefins, n-paraffins, isoparaffins, naphthenes, or aromatics. The reactions employed include absorption of olefins or olefins plus aromatics, hydrogenation of olefins, dehydrogenation of naphthenes and absorption of straight-chain compounds. Separation is carried out on a 2-metre didecyl phthalate column at 125°. The method has been successfully applied to the analysis of a synthetic sample and a commercial petrol. C. B. Baines

5090. **Determination of non-aromatic unsaturates in automobile exhaust by spectrophotometric titration.** P. P. Mader, K. Schoenemann and M. Eye (Air Pollution District, Los Angeles 13, Calif., U.S.A.). *Anal. Chem.*, 1961, **33** (6), 733–736.—The method, which depends on the high molar absorption of the free tribromide ion in the u.v. region, involves the absorption of the hydrocarbons from the gas in CCl_4, followed by spectrophotometric titration of suitable aliquots with small increments of 0·01N-bromide - bromate soln. (prep. described). The recovery of olefin ranged from 80 to 105% for compounds having 5 or more C atoms in the molecule and was ≃50% for C_4 olefins. The concn. range covered is from 25 to 1000 p.p.m. by vol. G. P. Cook

5091. **The aluminium - alizarin complex as a measure of Friedel - Crafts catalysts in paraffin hydrocarbon systems.** H. Pobiner (Esso Res. and Engng Co., Linden, N.J., U.S.A.). *Anal. Chem.*, 1961, **33** (6), 790–793.—The aluminium - alizarin complex is formed in the presence of an aq. NH_3 - thioglycollic acid buffer, which prevents interference from the excess of halogen acid. The soln. is heated at 57° to develop the red colour, and the extinction is measured at 508 mμ. The method is valuable for measuring the consumption of aluminium halide in Friedel - Crafts reactions. The 95% confidence limit is 2·9% at the 1·29% level of $AlBr_3$. G. P. Cook

5092. **Identification of components in paraffin wax by high-temperature gas chromatography and mass spectrometry.** E. J. Levy, R. R. Doyle, R. A. Brown and F. W. Melpolder (Atlantic Refining Co., Philadelphia, Pa., U.S.A.). *Anal. Chem.*, 1961, **33** (6), 698–704.—By the method described, 67 compounds present in a refined paraffin wax were identified. Mass spectrometry was used to assist in identification of the peaks, and the compounds separated by gas chromatography consisted of C_{22} to C_{32} homologues of n-paraffins, and 1-cyclopentyl, 1-cyclohexyl, 2 to 5-methyl and 1-phenyl compounds, and mono- and di-methyl cyclic compounds. Microcrystalline wax, asphaltene fractions and an aromatic extract were used as stationary phases, with a column temp. of ≃300° and a thermal-conductivity detector. The method can easily be developed into a routine procedure, and, with scaling up of apparatus, can be used to prepare pure components for physical testing procedures. C. B. Baines

5093. **Comparison of procedures for the determination of polycyclic aromatic hydrocarbons in [paraffin] waxes.** W. Lijinsky, C. R. Raha and J. Keeling (Chicago Med. Sch., Ill., U.S.A.). *Anal. Chem.*, 1961, **33** (6), 810–812.—Several procedures were compared. All required the concentration of the aromatic substance, usually by chromatography, and its separation from non-aromatic material. The individual polycyclic hydrocarbons were then determined in the appropriate fractions by various micro-methods. Several modifications were investigated. G. P. Cook

5094. **Determination of small amounts of water in insulating oil.** H. Kawinski (Firma Transformatorenwerk Lepper, Bad Honnef, Rhein, Germany). *Erdöl u. Kohle*, 1961, **14** (4), 271–275.—The Karl Fischer method with potentiometric dead-stop end-point detection has been applied to the determination of $\leqslant$1 p.p.m. of water in oil. The Karl Fischer reagent is diluted with methanol so that 1 ml $\equiv$ 0·5 mg of water. The apparatus (illustrated) is designed to exclude atmospheric moisture, and includes a tap-funnel from which the sample (50 g) is weighed into the titration vessel, where it is dispersed by magnetic stirring in 100 ml of methanol previously treated with Karl Fischer reagent. Since the end-point of the back-titration is not satisfactory, the determination is completed by adding an excess of methanol containing a known amount of water, and again titrating with Karl Fischer reagent. The coeff. of variation of duplicate determinations of known amounts (10 to 30 p.p.m.) of added water was $\pm$0·8%, and 8 to 10 determinations can be carried out per day. A. R. PEARSON

5095. **Methods for the analysis and testing of coal and coke. Part 16. Reporting of results.** British Standards Institution (2 Park Street, London, W.1). B.S. 1016: Part 16: 1961. 32 pp.—This publication contains sections on definitions of terms, the bases of reporting results for coal and coke and the interpretation of results.

5096. **Methods for the analysis of non-soapy detergent (NSD) products.** G. F. Longman and J. Hilton (Unilever Res. Lab., Port Sunlight, Cheshire). Monograph No. 1, Society for Analytical Chemistry, 1961. 30 pp.—A scheme is presented for the complete analysis of the organic and inorganic components of a detergent product. Details are given under the following main headings: determination of light-petroleum-soluble matter, ethyl ether-soluble matter and fatty acids; determination of active-matter content; detailed identification of active matter; determination of combined N, S and halogens; determination of inorganic constituents; interpretation of analytical results. S. M. MARSH

5097. **Analysis of volatile oils by chromatography on paper or plates. Application to some official essences.** R. Paris and M. Godon (Lab. de Mat. Méd., Fac. de Pharm., Paris, France). *Ann. Pharm. Franç.*, 1961, **19** (2), 86–93.—The conditions for satisfactory inverse-phase paper chromatography and chromatography on a silica layer between glass plates of volatile oils are investigated. *Procedures:* (*i*) *Inverse paper chromatography of untreated oils*—The paper is impregnated with ethylcellulose, nitrocellulose or 1-bromonaphthalene and the chromatogram is run between glass plates, because of the volatility of the compounds, with 40 to 50% aq. acetone or aq. tetrahydrofuran as developing solvent. Phenols are detected by 1% $FeCl_3$ soln., diazotised sulphanilic acid or *p*-nitroaniline, ketones by 2,4-dinitrophenylhydrazine, or by spraying with hydroxyammonium chloride from which HCl is liberated (changing the colour of bromophenol blue), and unsaturated substances are detected with fluorescein and bromine or saturated $SbCl_3$ in $CHCl_3$. (*ii*) *Inverse paper chromatography of mercurated oils*—The oils (particularly unsaturated ones) are treated with mercuric acetate and the chromatograms are run as in (*i*) on paper impregnated with 1-bromonaphthalene, using as developing solvent, acetone (60 ml) plus official ammonia (1·5 ml) plus water (38·5 ml) saturated with 1-bromonaphthalene. Detection is by spraying with ethanolic diphenylcarbazone. (*iii*) *Chromatoplate technique with a silica layer 0·5 mm thick*—Hexane, benzene or a 1:4 mixture of the two is used as developing solvent and the same detecting reagents are used. The application to a number of volatile oils is described. E. J. H. BIRCH

5098. **Analysis of cellulose acetobutyrate.** J. Franz (Inst. Chem. Tech. Plaste, Leipzig, Germany). *Plaste u. Kautsch.*, 1960, **7** (10), 493–494.—The sample is saponified with ethanolic alkali for 72 hr. in the cold, the pptd. cellulose is filtered off, washed and dried at 80°. The ethanol is then distilled off, the residue is dissolved in H_2O, alkali is removed on an ion-exchange column and the total acid is titrated with alkali. Equations have been derived for calculation of the acetyl and butyryl contents of the samples from the initial wt., the sap. value and the wt. of cellulose. L. A. O'NEILL

5099. **Identification of organotin stabilisers in poly(vinyl chloride) by thin-layer chromatography.** M. Türler and O. Högl (Inst. f. LebensmittChem., Univ., Bern, Switzerland). *Mitt. Lebensmitt. Hyg., Bern*, 1961, **52** (2), 123–130.—The organotin compounds are isolated from poly(vinyl chloride) by shaking the ethyl ether extract with aq. EDTA (disodium salt) followed by extraction with dithizone (in $CHCl_3$). Alternatively, the film may be soaked in an oil (*e.g.*, olive oil) and the organotin compounds adsorbed from the oil on to activated silica gel. The mobile phase is either butanol - acetic acid (50 to 100: 1) saturated with water or water - butanol - ethanol - acetic acid (20: 10: 10: 1), and the spots are revealed with dithizone or diphenylcarbazone. Dibutyl-, dioctyl- and dibenzyl-tin salts have been separated and identified, and mixtures of dialkyl- and trialkyl-tin compounds have been separated. R. CLARKE

5100. **Polarographic determination of methacrylate.** V. N. Dmitrieva and E. Yu. Novik (Kharkov Dental Materials Works). *Zavod. Lab.*, 1961, **27** (4), 395–396.—The sample (0·7 to 0·8 g) of plastic or plastic emulsion powder is dissolved in 10 ml of benzene and 4 vol. of methanol are added. When the polymer has settled, an aliquot of the soln. is added to the basal soln. (0·02N-tetramethylammonium iodide in 92% methanol), and a polarogram covering the $E_{\frac{1}{2}}$ (−1·79 V) of methyl methacrylate is obtained after removal of O by H. The presence of residual styrene has no effect. G. S. SMITH

5101. **Determination of acrylate and maleate esters in polymers by combined Zeisel and gas-chromatographic analysis.** D. L. Millar, E. P. Samsel and J. G. Cobler (Special Services Lab., The Dow Chemical Co., Midland, Mich., U.S.A.). *Anal. Chem.*, 1961, **33** (6), 677–680.—In the method described, the total alkoxyl groups are first determined by a modified Zeisel method and then the various alkoxyl groups, after conversion into the corresponding alkyl iodides, are collected in a cold trap and separated on a column (9 ft. × 0·25 in.) of di-(2-ethylhexyl) sebacate on firebrick at 70°. The method has been used for the determination of ester plasticisers in various plastic formulations and in mixtures. C. B. BAINES

5102. **Analysis of pyrolysates of polystyrene and poly(methyl methacrylate) by gas chromatography.** F. A. Lehmann and G. M. Brauer (Dental Res. Sta., Nat. Bur. of Standards, Washington 25, D.C., U.S.A.). *Anal. Chem.*, 1961, **33** (6), 673–676.—A study is made of the volatile products resulting from pyrolysis of polymers at temp. ranging from 425° to 1125°. Separation of the products is obtained on columns of Apiezon L, Chlorowax 70, or dinonyl phthalate on firebrick or silica gel. The method can be applied to other polymers, and yields valuable information with respect to the structure and constituents. The pyrolysis chamber is illustrated. C. B. BAINES

5103. **Amperometric determination of the total content of phenolic groups in epoxide resins.** V. I. Ginzburg and L. M. Flegontova. *Zavod. Lab.*, 1961, **27** (4), 392–394.—With a basal electrolyte of 3·3M-dimethylformamide and 5M-methanol in 0·5M-HCl and a rotating platinum electrode at 0·1 to 0·3 V, phenolic groups are titrated amperometrically with 0·05N-$KBrO_3$ - KBr.
G. S. SMITH

5104. **Detection of antioxidants in different varieties of polyethylene.** V. Cieleszky and F. Nagy (Inst. f. Ernährungswissensch., Budapest, Hungary). *Z. Lebensmitt Untersuch.*, 1961, **114** (1), 13–18.—*Procedure*—Remove dust from the granular material by a current of air. Add to the sample (5 g) anhyd. ethyl ether (50 ml) and set aside in the dark, with occasional shaking, for 24 hr. at room temp. Allow the filtered extract to evaporate at room temp., dry the residue in a desiccator, and then dissolve it in cyclohexane - ethanol (4:3) to give a concn. of a few mg per ml. Spectrophotometric examination of this soln. shows very distinct absorption minima at 250 mμ and peaks at 275 to 285 mμ when any one of the usual antioxidants is present; similar soln. obtained from antioxidant-free samples show a steady curvilinear decline over this range. P. S. ARUP

5105. **Spectrometric analysis of modified rosin.** V. S. Fikhtengol'ts (S. V. Lebedev Sci. Res. Inst. of Synthetic Rubber). *Zavod. Lab.*, 1961, **27** (4), 400–403.—The extinctions of the ethanol soln. are measured at 241 mμ (max. for abietic acid), 250 mμ (max. for isoabietic acid) and 276 mμ (max. for dehydroabietic acid), and the contents are calculated by means of formulae.
G. S. SMITH

5106. **Infra-red spectroscopy.** Chicago Society for Paint Technology (Chicago 23, U.S.A.). *Off. Dig. Fed. Soc. Paint Technol.*, 1961, **33**, 160 pp.—A comprehensive account is given of the use of infra-red spectroscopy in the paint field, covering the theory, the preparation of samples for analysis (as films, solutions, mulls or pressed discs), the care of cells, and procedures for qualitative and quantitative analysis. Reference spectra (195) of resins, oils, solvents, pigments and additives are given. (259 references.) L. A. O'NEILL

5107. **Detection of nitrocellulose [in paint].** H. F. Vollmann (Vollmann Bros. Ltd., Slough, Bucks., England). *J. Oil Col. Chem. Ass.*, 1961, **44** (5), 308–310.—It is shown that the diphenylamine test for the detection of nitrocellulose in paint gives erroneous results in the presence of certain resins. Changes in the composition of the reagents also affect the results. An improved method with fresh Griess - Ilosvay reagent (equal vol. of 1% sulphanilic acid and 0·1% 1-naphthylamine in 30% acetic acid) is described. As little as 0·1 g of paint scrapings can be used. *Procedure*—Paint is dried on a glass plate and ≃ 0·2 g is transferred to a basin covered with a thick filter-paper moistened with fresh reagent; 0·2 to 0·5 ml of conc. H_2SO_4 is added and the mixture is warmed to 50°. A pink colour appears on the paper if nitrocellulose is present.
E. S. LANE

5108. **Methods of testing synthetic rubber latices.** British Standards Institution (2 Park Street, London, W.1). B.S. 3397:1961. 9 pp.—Methods for sampling, total solids, coagulum, pH and surface tension are described.

5109. **Polarographic determination of dimethyl and dibutyl phthalates in propellents with use of zinc amalgam to overcome interference from nitroglycerine.** J. Townend and E. Macintosh (Naval Ordnance Inspection Lab., Caerwent, Chepstow, Mon., England). *Analyst*, 1961, **86**, 338–342.—The sample containing ≃500 mg of nitroglycerine is extracted in a Soxhlet apparatus for 7 hr. with ethyl ether. After removal of ether, the residue is dissolved in ethanol (25 ml), zinc amalgam (6 g) is added and the mixture is shaken mechanically for 1 hr. The liquid is filtered with suction through a Buchner funnel of specified type, the amalgam is washed with ethanol and the filtrate and washings are diluted with ethanol to 100 ml. To a 25-ml aliquot are added 10 ml of tetramethylammonium bromide soln. (20% v/v), 0·5 ml of a 1% ethanolic indicator soln. (cresol red and thymol blue, 1:3, v/v) and then aq. tetramethylammonium hydroxide soln. (≃0·5N) until a permanent change of colour occurs. After addition of 1 ml of aq. gelatin soln. (0·5%) the liquid is adjusted to 100 ml with ethanol, washed nitrogen equilibrated with water and ethanol is passed through, and the polarogram is recorded from −1·4 to −1·8 V *vs.* the mercury-pool anode. Standard polarograms are recorded with standard dimethyl or dibutyl phthalate added to a reduced nitroglycerine soln. Both sample and standard polarograms are corrected by means of the polarogram of a reduced nitroglycerine soln. *A. O. JONES

See also Abstracts—**4918**, Papers from I.U.P.A.C. Congress, Lisbon, 1956. **4944**, Cu and Zn 8-hydroxyquinolinates in textiles. **4977**, Red lead for paints. **5033**, Phosphorus in coal and coke. **5121**, Determination of ^{14}C. **5122**, Determination of ^{14}C and ^{32}P. **5144**, Diethyldithioacetals of monosaccharides. **5253**, Alkoxyphenols in vanillin. **5301**, Apparatus for pure CO_2 for N determinations.

4.—BIOCHEMISTRY
INCLUDING DRUGS, FOOD, SANITATION, AGRICULTURE

Biological fluids, animal and vegetable tissues

5110. **Use of the "Hersch cell" for the measurement of oxygen in biological material.** D. L. Dewey and L. H. Gray (Mt. Vernon Hospital, Northwood, Middx., England). *J. Polarographic Soc.*, 1961, **7** (1), 15–25.—An improved version of the Hersch cell (*Instrum. Practice*, 1957, **11**, 817, 937) is described, which is easier to construct and

can be water-jacketed. The response of the voltaic cell to a partial pressure of O in a gas stream may be measured directly as a current or as a reading on a fluxmeter. The zero drift of the cell is equivalent to a rate of change in the composition of the carrier gas of $\simeq 2 \times 10^{-6}$ parts of O per hr. and the sensitivity enabled 6×10^{-2} μl of O to be measured with 1% accuracy. Applications described include monitoring the O content of a flowing gas, measuring the permeability to O of various plastics, and measuring the O content of a liquid sample and the O content of respiring bacterial cell suspensions. Interferences are briefly discussed. G. RUSSELL

5111. **Method for determination of carbon monoxide in a gas phase applied to the determination of the carbon monoxide capacity of blood.** H. Dahlström (Lab. of Clin. Physiol., Med. Högskolan i Umeå, Sweden). *Scand. J. Clin. Lab. Invest.*, 1960, **12** (4), 396–401 (in English).—The method described is based on diffusion of the CO into oxygen and passage of the gas into an indicator tube filled with a gel containing a complex silicon - molybdenum compound $[H_8Si(Mo_6O_7)_6]$ and $PdSO_4$. The colour change is measured and compared with standards. Calibrations are needed only infrequently, and the precision is good in the range 0·1 to 20 p.p.m. P. PEDERSEN

5112. **The determination of deuterium in biological fluids.** H. L. Crespi and J. J. Katz (Argonne Nat. Lab., Argonne, Ill., U.S.A.). *Anal. Biochem.*, 1961, **2** (3), 274–279.—A micro-method by the use of the infra-red absorption peaks of water, at 1·44 and 1·66 μ, is described. Concn. are estimated from the difference in extinction between the HOH peak at 1·44 μ, and the HO^2H peak at 1·66 μ, or directly from the latter in the case of high concn. of ^2H. The method gives good results when applied directly to serum or clear urine samples. D. G. Moss

5113. **Rapid micro-method for the analysis of skeletal muscle for water, sodium, potassium, chloride and fat.** C. T. G. Flear and I. Florence (Queen Elizabeth Hosp., Birmingham, England). *Clin. Chim. Acta*, 1961, **6** (1), 129–135.—The complete analysis is made on one sample. Water is determined by drying slices of tissue (10 to 14 mg) in small aluminium pans for 30 min. at 105°. Fat is extracted by treating the dried tissue with light petroleum for 10 min. The tissue is then dried and reweighed. The electrolytes are extracted by allowing the dried fat-free tissue slices to stand in 5 ml of 0·1N-HNO_3 for 1 hr. Sodium and K are determined by flame photometry and Cl by the method of Saunderson (*Biochem. J.*, 1952, **52**, 502). H. F. W. KIRKOATRICK

5114. **Micro-methods for the determination of inorganic components (chlorine, potassium, sodium, calcium, magnesium, iron, copper) in biological material.** K. Beyermann and K. Cretius (Inorg. Chem. Inst., Univ., Mainz, Germany). *Clin. Chim. Acta*, 1961, **6** (1), 113–124 (in German).—The methods are described in their application to dried muscle. Chlorine is determined by a modification of the method of Schöniger (*Anal. Abstr.*, 1955, **2**, 1816). Pre-treatment of the sample by wet digestion before the determination of the other constituents is described. Sodium and K are determined by flame photometry, Ca with EDTA and murexide, the titration being followed photometrically, Fe and Mg as 8-hydroxyquinolinates, and Cu with Na diethyldithiocarbamate. H. F. W. KIRKPATRICK

5115. **The loss of sodium and potassium during the dry-ashing of animal tissue.** E. L. Grove, R. A. Jones and W. Mathews (School of Chem., Univ. Alabama, University, Ala., U.S.A.). *Anal. Biochem.*, 1961, **2** (3), 221–230.—The maximum temp. at which animal tissue could be dry-ashed for 24 hr. without appreciable loss of Na and K was found to be 550°. In the 800° to 900° range, approx. 90% of the K and 40 to 70% of the Na are lost from blood and serum samples. D. G. Moss

5116. **A new method for determination of potassium in serum.** E. Bladh and P. Gedda (Dept. for Rheumatic Diseases, Univ. Hospital, Lund, Sweden). *Scand. J. Clin. Lab. Invest.*, 1960, **12** (3), 274–276.—The potassium is precipitated with Na tetraphenylborate and the excess of the reagent is determined by turbidimetric titration with cetyltrimethylammonium bromide (cetrimide). P. PEDERSEN

5117. **Complexometric determination of potassium in urine.** A. Holasek and M. Pecar (Med. Chem. Inst., Univ., Graz, Austria). *Clin. Chim. Acta*, 1961, **6** (1), 125–128 (in German).—The urine is treated with trichloroacetic acid to remove any protein, formaldehyde is added to prevent interference by NH_3 and the K is pptd. with cobaltinitrite. The K is titrated with EDTA, with the Cu and 1-(2-pyridylazo)-2-naphthol complex as indicator. H. F. W. KIRKPATRICK

5118. **Separation of calcium and magnesium in biological fluids using a cation-exchange resin.** D. E. Stevenson and A. A. Wilson (Sch. of Vet. Med., Univ. of Cambridge, England). *Clin. Chim. Acta*, 1961, **6** (2), 298–299.—Zeo-Karb 225 (20 NC) is extracted several times with hot 6N-HCl until the extract is free from Fe, washed to remove excess of acid and packed into short polyethylene tubes to form columns 8 cm × 0·7 cm. The columns are washed with about 10 bed-vol. of 15% KCl soln. to convert the resin to the K^+ form and then with H_2O to remove Cl^-. Plasma or serum is diluted with 2 or 3 vol. of normal saline and applied to the column, and the column is washed with saline (urine, tissue extracts and ash soln. need no dilution and the column can be washed with H_2O). Elution is with 15% KCl soln. at a flow rate of 6 to 10 ml per hr. Magnesium is recovered from the 3rd to the 8th ml, inclusive, and Ca from the 13th to the 25th ml. The fractions are titrated with EDTA (disodium salt), with Solochrome black T (C.I. Mordant Black 11) as indicator. H. F. W. KIRKPATRICK

5119. **Ion-exchange separation of calcium and strontium. Application to determination of total strontium in bone.** M. A. Wade and H. J. Seim (Univ., Reno, Nevada, U.S.A.). *Anal. Chem.*, 1961, **33** (6), 793–795.—The routine determination of Sr in the presence of up to 20,000 times its wt. of Ca, *e.g.*, in bone ash, may be carried out by flame photometry (oxy-hydrogen flame at 460·7 mμ) after separation of most of the Ca. The separation procedure described is based on the difference in the stability constants of the EDTA complexes of Ca and Sr. *Procedure*—Bone ash (1 g) is dissolved

in HCl, evaporated to dryness and redissolved in a 2% soln. of the ammonium salt of EDTA (pH 5·3) (200 ml). The pH is adjusted to 4·8 and the soln. is passed through a column of Dowex 50-X8 (NH_4^+ form). Calcium is eluted with the ammonium EDTA soln., the column is washed with water, residual alkali metals, Mg^{2+} and NH_4^+ are eluted with 0·75N-HCl, and Sr is then eluted with 3N-HCl. G. S. ROBERTS

5120. **High-sensitivity detection of naturally occurring radio-carbon. I. Chemistry of the counting sample.** M. A. Tamers, J. J. Stipp and J. Collier (Dept. of Chem., Univ. of Texas, Austin, Texas, U.S.A.). *Geochim. et Cosmoch. Acta*, 1961, **24** (3–4), 266–276.—The use of radiation detection techniques greatly increases the sensitivity of radio-carbon dating. For use with a liquid-scintillation spectrometer, the sample to be dated is used to produce benzene, as benzene contains 92% of C and shows no scintillation-quenching properties. Details are given for the combustion of the sample, collection of the CO_2 in aq. NH_3 and precipitation of $SrCO_3$ by the addition of $SrCl_2$. The dried carbonate is mixed with magnesium powder, and strontium carbide is produced by ignition in an evacuated stainless-steel tube. Reaction of the carbide with water generates acetylene, which is led into a circuit containing traps cooled with solid CO_2 and a pyrolysis tube at 600°, thus polymerising the acetylene to benzene, which is then purified by distillation. R. A. HOWIE

5121. **Determination of carbon-14 and tritium in biological samples by Schöniger combustion and liquid-scintillation techniques.** R. G. Kelly, E. A. Peets and D. A. Buyske (Lederle Lab., Pearl River, N.Y., U.S.A.). *Anal. Biochem.*, 1961, **2** (3), 267–273.—Samples of tissue or biological fluid, up to 200 mg dry-weight, were placed in small cups of cellulose film, and allowed to dry. Combustion under oxygen was carried out in modified Schöniger flasks, constructed from 2-litre filter-flasks, connected at the side arm with a stopcock and a 20-ml reservoir. After combustion, the CO_2 was absorbed in the hydroxide prepared from Hyamine 10-X (a quaternary ammonium salt) or ethanolamine soln., which was then dissolved in a toluene soln. of a phosphor (PPO and POPOP), for determination of ^{14}C by liquid scintillation. Tritiated water was collected as ice, and dissolved in a toluene - ethanol soln. of the phosphor.

D. G. Moss

5122. **Determination of carbon-14 and phosphorus-32 in animal tissue and blood fractions by the liquid-scintillation method.** H. G. Badman and W. O. Brown (Dept. of Agric. Chem., Queen's Univ., Belfast, N. Ireland). *Analyst*, 1961, **86**, 342–347.—In a study of the influence of the vol. of scintillator, sample and the blending agent Hyamine 10-X [benzyldimethyl-2-methyl-4-(1,1,3,3-tetramethylbutyl)phenoxyethoxyethylammonium chloride monohydrate] on the efficiencies of counting of water-sol. sources of ^{32}P, max. efficiency (81%) for an aq. soln. of ^{32}P was achieved with 3 ml of scintillator compared with a max. efficiency of 65% with 8 ml for ^{14}C (Brown and Badman, *Biochem. J.*, 1961, **78**, 571). This is probably attributable to the higher energy of the β-emission from ^{32}P. With lipid material (an ethereal extract of oviduct tissue containing ^{32}P) neither the vol. of scintillator nor the use of dioxan as blending agent with lipid extract and blood sera had any effect on the efficiency of the counting. The counting efficiency (99%) of ^{32}P-labelled lipid extracts was determined by comparing the counting rates before and after converting the P into the inorganic state. The counting efficiency (81%) of ^{14}C-labelled extracts of oviduct tissue was determined by the recovery of activity added as [1-^{14}C]stearic acid, and similar results were obtained in the recovery of [^{14}C]benzoic acid from blood sera. The activities of fractions of labelled blood sera and oviduct tissue were together equal to the determined activity of the respective whole samples.

A. O. JONES

5123. **A modified Conway technique for the determination of carbonates [in blood] with titration carried out during magnetic stirring in a carbon dioxide-free atmosphere.** I. Holm-Jensen (Pharmacol. Inst., Univ., Aarhus, Denmark). *Scand. J. Clin. Lab. Invest.*, 1960, **12** (3), 269–273.—Precautions for excluding CO_2 from the apparatus are described. Before and after the analytical manipulations, the Conway units are washed with CO_2-free air. Magnetic stirring is applied. Samples of 0·1 ml containing 25 millimoles of carbonate per litre can be determined within ±0·8%.

P. PEDERSEN

5124. **Formation of artefactual ammonia in blood by action of alkali. Its significance for the measurement of blood ammonia.** J. G. Rheinhold and C. C. Jung (William Pepper Lab. of Clin. Med., Philadelphia, Pa., U.S.A.). *Clin. Chem.*, 1961, **7** (1), 54–69.—It is shown that NH_3 is formed by decomposition of protein by the action of alkali during blood analysis. This effect is avoided if the preformed NH_3 is liberated by a buffer soln. prepared by adding 96 g of $Na_2B_4O_7.10H_2O$ and 19·6 g of NaOH to approx. 800 ml of H_2O and boiling for 15 min. with stirring. After cooling, the soln. is made up to 1 litre with H_2O, when the pH should be 10·8 ±0·2. Separation of borate crystals may be disregarded.

H. F. W. KIRKPATRICK

5125. **Determination of bromide in blood.** H. V. Street (Dept. of Forensic Med., Univ. of Edinburgh, Scotland). *Clin. Chim. Acta*, 1960, **5** (6), 938–941.—To a mixture of blood (2 ml) and H_2O (2 ml) add slowly with shaking 20% trichloroacetic acid soln. (10 ml), shake vigorously, allow to stand for 5 min. and filter. Shake the filtrate (9 ml) with ethyl ether (5 ml), remove the aq. layer (7 ml), add 6N-H_2SO_4 (2 ml) and a few crystals of $KMnO_4$ and then cyclohexane (3 ml), shake vigorously for 20 to 25 sec. Centrifuge, and read the extinction of the cyclohexane layer at 405 mμ against a blank with H_2O in place of blood. Prepare a standard curve with NaBr soln. (0 to 200 mg per 100 ml) treated in the same way as blood.

H. F. W. KIRKPATRICK

5126. **Automatic titration with direct read-out of chloride concentration.** E. Cotlove and H. H. Nishi (Nat. Heart Inst., Bethesda, Md., U.S.A.). *Clin. Chem.*, 1961, **7** (3), 285–291.—An auxiliary unit for use with the automatic coulometric - amperometric titrator previously described (Cotlove *et al.*, *Anal. Abstr.*, 1959, **6**, 272) provides a direct reading of the Cl^- concn. in milli-equiv. per litre on a digital register and requires 0·1 ml of sample for the determination. H. F. W. KIRKPATRICK

5127. **Inorganic phosphate and alkaline phosphatase determinations according to Raabe.** O. Meulemans (Dept. of Pediatrics, State Univ., Utrecht, The Netherlands). *Clin. Chim. Acta*, 1961, **6** (1), 145–146.—Statistical comparison of Raabe's method (*Anal. Abstr.*, 1955, **2**, 2243) with the method of Briggs (*J. Biol. Chem.*, 1922, **53**, 13) for inorganic phosphate, and of Jenner and Kay (*Brit. J. Exp. Path.*, 1932, **13**, 22) for phosphatase, showed no significant difference in the results. The method, which has certain advantages over existing procedures, thus has an acceptable validity. H. F. W. KIRKPATRICK

5128. **Micro-determination of serum phosphate and phospholipids.** E. Baginski and B. Zak (Sinai Hosp., Detroit, Mich., U.S.A.). *Clin. Chim. Acta*, 1960, **5** (6), 834–838.—*Serum phosphorus*—Add the serum (0·1 ml) to 10% trichloroacetic acid (2 ml) with shaking, mix and allow to stand for 10 min. Centrifuge, and to 1 ml of supernatant fluid add 10% ascorbic acid soln. (0·2 ml), 1% aq. ammonium molybdate (0·5 ml) and arsenite - citrate reagent [Na citrate (10 g) and $NaAsO_2$ (10 g) are dissolved in H_2O, glacial acetic acid (10 ml) is added and the soln. is diluted to 500 ml with H_2O] (1 ml). After 15 min. read at 700 mμ against a blank. Prepare standards in 10% trichloroacetic acid for calibration. A method for serum phosphatase is described with Na β-glycerophosphate as substrate and determination of the liberated phosphate. Serum phospholipids are extracted with Bloor's reagent and the residue after evaporation is digested with HNO_3 (2 ml) by heating to dryness. The phosphorus is determined by dissolving the residue in 10% trichloroacetic acid (2 ml) and developing the colour as for serum phosphorus.
H. F. W. KIRKPATRICK

5129. **Turbidimetric determination of inorganic sulphate in serum, plasma and urine.** F. Berglund and B. Sörbo (Dept. of Pharmacol., Karolinska Inst., Stockholm, Sweden). *Scand. J. Clin. Lab. Invest.*, 1960, **12** (2), 143–153.—A $BaCl_2$ - gelatin reagent containing preformed $BaSO_4$ particles, which function as nuclei for the pptn., is described. Heparin and thiosulphate interfere.
P. PEDERSEN

5130. **Chemical concentration and spectrographic determination of certain minor trace metals in plant material.** A. Strasheim and D. J. Eve (National Phys. Res. Lab., S. African Council for Sci. and Ind. Res., Pretoria). *J. S. Afr. Chem. Inst.*, 1961, **14** (1), 1–15.—Results obtained from the analysis of the leaves of peach, bean, beet, plum and tobacco are reported. The separation of Zn, Co, Ni, Pb, Ti, V and Mo from Fe and other constituents by successive extractions with $CHCl_3$ from a soln. at predetermined pH values is described. Dithizone, 8-hydroxyquinoline and cupferron are used as complexing agents. The combined extracts are evaporated on to graphite powder, loaded into a graphite electrode, burned in an interrupted d.c. arc and spectrographed. The coeff. of variation calculated from analyses of sixteen aliquots of a soln. of plant material were—Zn, 3·6%; Co, 14·5%; Ni, 6·4%; Pb, 22%; Ti, 11·6%; V, 11·3%; and Mo, 10·6%. E. G. CUMMINS

5131. **Determination of microgram amounts of tin in animal and plant material by the dithiol method.** K. Bürger (Farbwerke Hoechst A.-G., Gendorf Obb, Germany). *Z. LebensmittUntersuch.*, 1961, **114** (1), 10–13.—The procedure described is a modification of the method of Jantsch *et al.* (*Ibid.*, 1948, **128**, 451). After the wet combustion (by H_2SO_4 plus HNO_3) of the sample (containing 5 to 40 μg of Sn) to destroy organic matter, and the elimination, by boiling with water, of HNO_3, hydrazine sulphate (1 to 2 g) is added, and the mixture is boiled until it reaches a temp. of 160°. A mixture of conc. HCl (15 ml) and HBr (7 ml) is then added (at first dropwise), and the mixture is distilled for 15 to 20 min. at 145° to 160°, the distillate, containing the Sn as $SnCl_4$, being collected in a flask containing water. The distillate is evaporated to fumes with H_2SO_4 and HNO_3 to remove HBr, Br and, finally, HNO_3, diluted with water (7 ml), and tested by adding 1·5% benzenesulphonate reagent (0·5 ml), followed by the thioglycollic acid (1 drop) and dithiol reagent (0·5 ml, prep. described). The colour is developed at 50° for 5 min., and the extinction is then measured at 530 mμ. The smallest detectable amount of Sn is 5 μg per 100 g. The error is within ±10%.
P. S. ARUP

5132. **Polarographic determination of selenium in urine.** A. G. Faulkner, E. C. Knoblock and W. C. Purdy (Div. of Nuclear Med. and Chem., Walter Reed Army Inst. of Res., Washington, D.C., U.S.A.). *Clin. Chem.*, 1961, **7** (1), 22–29.—Evaporate the urine (10 ml) to dryness, dissolve the residue in conc. HNO_3 (10 ml), heat until most of the HNO_3 is evaporated, add H_2SO_4 (1·5 ml) and heat to white fumes. Remove from the heat, add 30% H_2O_2 (1 ml) and heat to destroy excess of H_2O_2. Dilute the soln. to 50 ml with H_2SO_4 to give a sample soln. 3M in H_2SO_4. Remove air with N and record the polarogram; the diffusion current of the first wave is in linear relationship with the Se concn. Prepare standard soln. by dissolving pure Se in conc. HNO_3, add H_2SO_4, heat to fuming, add 30% H_2O_2 and heat again. Dilute this soln. as required to give 0·5 to 14 μg per ml of Se in 3M-H_2SO_4. Urine loses Se when allowed to stand, but if the urine is acidified to M in HNO_3 the Se content is retained for 4 days.
H. F. W. KIRKPATRICK

5133. **Determination of iron in biological materials by the *p*-amino-*NN*-dimethylaniline reaction.** G. Ceriotti and L. Spandrio (Centre for the Study of Tumours, Busto Arsizio, Italy). *Clin. Chim. Acta*, 1961, **6** (2), 233–236 (in English).—The material is digested with HNO_3, H_2SO_4 and H_2O_2. The acids are completely removed and the residue is ashed. The colour reaction is applied as follows. To the ashed residue add 2N-H_2SO_4 (2 ml) and heat the tube for 15 min. in a boiling-water bath, cool, add 80% Na acetate soln. (1 ml) and H_2O (1 ml). Shake and add 0·1% *p*-amino-*NN*-dimethylaniline hydrochloride soln. in 95% ethanol (1 ml) and mix thoroughly. Read the colour immediately at 515 mμ against a blank.
H. F. W. KIRKPATRICK

5134. **Direct determination and binding capacity of serum iron.** A. L. Levy and P. Vitacca (St. Vincent's Hosp., New York, N.Y., U.S.A.). *Clin. Chem.*, 1961, **7** (3), 241–248.—*Serum iron*—To the serum (0·6 ml) add H_2O (0·24 ml), phosphate buffer - ascorbic acid reagent (0·24 ml) and 2,2′:6′,2″-terpyridyl soln. (0·12 ml). Prepare a serum blank by mixing serum (0·6 ml), H_2O (0·36 ml) and buffer reagent (0·24 ml), a terpyridyl blank by replacing the serum in the test mixture with H_2O, and a

H_2O blank by replacing the serum in the serum blank with H_2O. Incubate the tubes at 45° for 20 min. and read at 552 mμ against H_2O. Prepare standards from a soln. containing 3 μg of Fe per ml using 0·1, 0·2 and 0·3 ml diluted to 0·84 ml with H_2O and proceeding as described above. Plot a calibration curve after subtracting the terpyridyl blank from the standard readings. Subtract the H_2O blank from the (serum blank + terpyridyl blank) and subtract this total blank from the test reading; read the concn. of Fe from the graph. *Unsaturated iron binding capacity (UIBC)*—To the serum (0·6 ml) add M-Tris buffer adusted to pH 8·5 with HCl (0·24 ml) and a standard soln. containing 20 μg of Fe per ml (0·12 ml), incubate at 45° for 10 min., add terpyridyl soln. (0·12 ml) and 1% metol - 3% $NaHSO_3$ (0·20 ml) and incubate for a further 20 min. Read at 552 mμ. A blank containing H_2O in place of serum is run at the same time. Subtract the blank reading from the test and then subtract the serum blank obtained in the serum-Fe determination; read the Fe content from the graph. The phosphate - ascorbic acid buffer reagent is prepared by mixing equal vol. of M-H_3PO_4 and M-Na_2HPO_4, adding 1 g of ascorbic acid to 100 ml of the mixture and adjusting to pH 5·0 with 2N-NaOH (approx. 6 ml); this soln. is stable for 1 week. The terpyridyl soln. is prepared by dissolving 100 mg in 4 ml of absolute ethanol, diluting to 40 ml with H_2O, adding 0·2N-HCl dropwise to clear the milky suspension and diluting to 100 ml (pH 4·3). Normal ranges by this method are, serum Fe, 44 to 155 mg per 100 ml, and UIBC, 150 to 336 mg per 100 ml.

H. F. W. KIRKPATRICK

5135. **X-ray spectroscopy in the clinical laboratory. IV. Phosphorus. Total blood iron as a measure of haemoglobin content.** S. Natelson and B. Sheid (Dept. of Biochem., Roosevelt Hosp., New York, U.S.A.). *Clin. Chem.*, 1961, **7** (2), 115–129.—The technique consists in placing samples of serum directly on to confined spots on paper, drying and passing through the X-ray field. Results for total P in serum and total Fe in whole blood showed no significant difference from those given by wet-ashing procedures, except that the total Fe is more precisely determined by wet-ashing. Haemoglobin levels determined by X-ray spectroscopy may have greater clinical significance, as no interference is experienced from bilirubin, leucocytes, red-cell debris or lipids, which interfere with the colorimetric method. Practical details of the method are given.

H. F. W. KIRKPATRICK

5136. **Identification of barbiturates by paper chromatography.** J. Fischl and S. Segal (Assaf Harofe Govt. Hosp., Zrifin, Israel). *Clin. Chem.*, 1961, **7** (3), 252–256.—The material (50 to 100 ml) is adjusted to pH 10 to 11 with 10% NaOH soln. and extracted with ethyl ether (50 ml). The ether extract is discarded and the aq. soln. is neutralised with HCl; 2 to 3 g of activated charcoal is added, mixed, set aside for 10 min. and filtered. The charcoal is dried, and the adsorbed barbiturate is extracted with ethyl ether - light petroleum (1:1) (40 ml) and filtered into a dish; the solvent is removed at 50° to 60°. The residue is dissolved in $CHCl_3$ (4 ml) and the soln. (0·1 ml) is applied to Whatman No. 1 paper for ascending chromatography. Details of various solvent systems and R_F values are given. The barbiturates are revealed by spraying the paper with 1% NaOH - 1% salicylamide in ethanol, drying and viewing under u.v. light, when they appear as dark violet spots on a brightly fluorescing background.

H. F. W. KIRKPATRICK

5137. **Method for the estimation of phenothiazine derivatives in urine and blood.** C. A. Hetzel (Wyeth Lab. Inc., Philadelphia, Pa., U.S.A.). *Clin. Chem.*, 1961, **7** (2), 130–135.—To the sample (5 ml) add 50% NaOH soln. (0·5 ml) and ethyl ether (30 ml), stopper the container, shake for 5 min. and centrifuge at 2000 r.p.m. for 1 min. Transfer 25 ml of the ether to a 50-ml tube, add 0·1N-H_2SO_4 (2 ml), stopper, shake and centrifuge as before. Discard the ether and remove ether completely from the aq. soln. by immersing the tube in H_2O at 65° under reduced pressure for 1 min. and then in boiling H_2O for 5 min. Add HNO_2 (4 ml) down the sides of the tube and heat in boiling H_2O for 20 min. (10 min. only for promethazine). Cool the tube to room temp. Read at 370 mμ for perphenazine, prochlorperazine, thiopropazate and chlorpromazine, or at 380 mμ for promazine and its metabolites and promethazine, against a blank prepared by heating and cooling similarly 1·3 ml of 0·1N-H_2SO_4 and 4 ml of HNO_2. Evaluate the reading from standard curves for these substances in the range 0 to 100 μg.

H. F. W. KIRKPATRICK

5138. **Determination of *N'N'*-anhydrobis-(β-hydroxyethyl)biguanide [*N*-(guanidinoformimidoyl-morpholine] hydrochloride in urine.** T. L. Flanagan, E. K. Horikawa and E. J. Van Loon (Smith, Kline and French Lab., Philadelphia, Pa., U.S.A.). *Anal. Biochem.*, 1961, **2** (3), 231–236.—A spectrophotometric method based on the difference in extinction at 237 mμ (maximum for the drug) and 285 mμ (minimum) is presented. Interference from other u.v. absorbing substances in urine was overcome by using as blank a portion of urine from which the drug had been removed by treatment with hypobromite. Pigments and protein are removed by a preliminary treatment with $Ba(OH)_2$ and $ZnSO_4$, followed by centrifuging and passage through a column of Dowex-1 resin. The resin column was washed with 0·1N-NaOH, which was added to the percolate. The blank was prepared by mixing 10 ml of the combined percolates (≡ 0·3 ml of urine) with 0·2 ml of satd. Br water, followed after 10 min. by 0·1 ml of 0·1N-$Na_2S_2O_3$. The mixed soln. was passed again through the resin column to remove Br^- and BrO^- which would otherwise interfere by absorption of u.v. light.

D. G. Moss

5139. **Method for determination of phenylalanine mustard and related alkylating agents in blood.** O. Klatt, A. C. Griffin and J. S. Stehlin (Dept. Biochem., Univ., Houston, Tex., U.S.A.). *Proc. Soc. Exp. Biol. & Med.*, 1960, **104** (4), 629–631.—To 1-ml aliquots of plasma from heparinised blood samples are added 0·1 ml of 5% acetic acid, 2·9 ml of water and 0·5 ml of 5% 4-(*p*-nitrobenzyl)pyridine in ethyl methyl ketone. The mixture is shaken, and then heated at 80° for 15 min. After cooling in ice and centrifuging, 2 ml of the supernatant liquid is mixed with 2 ml of triethylamine soln. (50% in acetone), and the extinction of the resulting purple soln. is read against water at 565 mμ. The Beer - Lambert law is obeyed over a range of 0·0 to 0·2 μmole of alkylating agent per ml. The soln. before addition of the colour reagent is stable for 1 hr. at room temp., but the colour fades rapidly

and must be measured immediately. For reproducible results, all conditions must be fully standardised. The reactions of *NN*-bis-(2-chloroethyl)phenylalanine (**I**), 5-[bis-(2-chloroethyl)-amino]uracil (U8344) (**II**), and *NN*-bis-(2-chloroethyl)methylamine (HN2) (mustine) (**III**) with blood, as indicated by a decreasing colour value with time, were investigated. It is shown that **I** and **II** have a similar action on blood, whereas **III** differs in its action. D. B. PALMER

5140. **Determination of *NN'*-diphenyl-*p*-phenylenediamine [DPPD] [*p*-dianilinobenzene] in animal tissues.** A. Saari Csallany and H. H. Draper (Div. Animal Nutrition, Univ. Illinois, Urbana, U.S.A.). *Proc. Soc. Exp. Biol. & Med.*, 1960, **104** (4), 739–742.—Approximately 10 g of tissue (liver or skeletal muscle) is ground with anhyd. Na_2SO_4 or with Na_2SO_4 and acid-washed sand, until dry, and the mixture is extracted with acetone overnight, at room temp., in darkness. The acetone extract is filtered, the residue is washed several times, and the total filtrate is reduced to 30 to 40 ml *in vacuo* at 40° under N. To remove the lipids, the extract is cooled in a solid CO_2 - acetone bath at −70° for 30 min., after which the ppt. is filtered off at the same temp. The residue is washed with cold acetone (5 × 10 ml), and the filtrate is evaporated to dryness *in vacuo* at 40° under N. The residue is dissolved in a suitable vol. of anhyd. ethanol. To 2-ml aliquots of this soln. is added 1 ml of alcoholic $FeCl_3$ soln. (0·1 g per 100 ml), and, after mixing, the extinction of the soln. is read at 700 mμ, against a reagent blank. Sample blanks are also measured to correct for any pigmentation of the tissue extracts. Beer - Lambert's law is obeyed over a range of 6 to 180 μg per 3 ml of soln. The blue - green pigment is stable for approx. 15 min.; the extraction and estimation should both be performed in a dim light. Quantitative recoveries of DPPD are reported. D. B. PALMER

5141. **Diagnostic compositions [for detecting conditions of abnormality in biological fluids].** Miles Laboratories, Inc. Brit. Pat 867,192, date appl. 11.12.58; U.S.A., date appl. 16.12.57.—A combination diagnostic composition is provided with which two or more tests may be made on a biological fluid simultaneously. The various portions of a carrier impregnated with different reagents are separated by barriers of ethylcellulose, polystyrene or rosin, which are impervious to aq. soln. Diagnostic indicators are given for glucose, albumin, ketone bodies and pH. J. M. JACOBS

5142. **Measurement of glucose in blood, serum and plasma by means of a glucose oxidase - catalase enzyme system.** F. W. Sunderman, jun., and F. W. Sunderman (Jefferson Med. Coll., Philadelphia, Pa., U.S.A.). *Amer. J. Clin. Path.*, 1961, **36** (1), 75–91.—The H_2O_2 formed when glucose is oxidised to gluconic acid by the action of glucose oxidase is induced by the action of catalase to oxidise methanol to formaldehyde, which is subsequently determined by the chromotropic acid reaction. Whole blood, serum or plasma (0·1 ml), followed by 0·5% $ZnSO_4.7H_2O$ soln. (1·9 ml), is added to 0·3% $Ba(OH)_2$ soln. (2·0 ml) and the mixture is centrifuged. Protein-free supernatant liquid (0·5 ml), glucose standard (30 μg per ml) (0·5 ml) and water (blank) (0·5 ml) are placed in three Pyrex-glass tubes (18 mm × 150 mm) and to each tube are added methanol (0·5 ml) and glucose oxidase in citrate - phosphate buffer (0·5 ml). (The buffer, pH 5·6, is prepared by diluting 290 ml of 0·2M-Na_2HPO_4 to 500 ml with 0·1M-citric acid; the optimum concn. of the glucose oxidase depends on the purity of the enzyme preparation used. Commercially available glucose oxidase preparations usually contain sufficient catalase activity to support the coupled methanol oxidation.) After incubating the tubes at 37° for 60 min. or 47° for 40 min., chromotropic acid reagent (4·0 ml) is added, and the tubes are then capped with large marbles and placed in a boiling-water bath for 6 min. [The chromotropic acid reagent is prepared by dissolving 0·5 g of 4,5-dihydroxy-2,7-naphthalenedisulphonic acid (disodium salt) in a few ml of water and adding 175 ml of conc. H_2SO_4 cautiously, with constant cooling in an ice bath, the vol. being made up to 250 ml with water.] After cooling the soln. to room temp. the extinctions are read at 575 mμ and the glucose concn. (mg per 100 ml) is then (extinction of sample/extinction of standard) × 120. An emergency procedure is also described in which the colour is developed after an accurately timed period (10 min.), but before the oxidation has gone to completion. Venous blood glucose (in 38 normal fasting persons) averaged 80 mg per 100 ml (standard deviation ±6·6 mg). Results averaged 9% lower than those obtained by the Nelson - Somogyi copper reduction procedure, and 7% lower than those obtained with an automated ferricyanide reduction technique. R. A. BRENAN

5143. **Thin-film chromatography. VI. Trace analysis of sugar mixtures on Kieselguhr-G films.** E. Stahl and U. Kaltenbach (Bot. Inst. Saarland Univ., Saarbrücken, Germany). *J. Chromatography*, 1961, **5** (4), 351–355 (in German).—Disperse 60 g of Kieselguhr-G (Merck) in aq. Na acetate soln. (0·02M) (60 ml). This is enough to coat 5 plates, 20 cm × 20 cm. Dry the coated plates for 30 min. at 100° and mark the origin line 1·5 cm from the lower edge. The solvent consists of ethyl acetate (65 ml) and isopropyl alcohol - H_2O (2:1) (35 ml), freshly prepared. Dissolve the sugars in pyridine, apply to the film and allow the solvent to run for ≃30 min. Detect the sugars by spraying each film with 10 ml of a reagent consisting of 95% ethanol (9 ml), conc. H_2SO_4 (0·5 ml) and anisaldehyde (0·5 ml). Heat for 5 to 10 min. at 90° to 100°. R_F values and colours are listed for 13 sugars. The minimum amount of sugar detectable (≃0·05 μg) is about one-hundredth that on paper. G. RUSSELL

5144. **Paper chromatography of diethyldithioacetals of 2-amino-2-deoxyhexoses, uronic acids and neutral monosaccharides.** K. Onodera and Y. Morisawa (Coll. of Agric., Univ., Kyoto, Japan). *Anal. Biochem.*, 1961, **2** (3), 263–266.—The diethyldithioacetals (diethylmercaptals) of uronic acids could be separated from those of *N*-acetylhexosamines by paper chromatography, with isopropyl alcohol - pyridine - acetic acid - water (8:8:1:4) as solvent system, and paper previously treated with borate buffer (pH 8) and dried. Suitable solvent systems for separation of the diethylmercaptals of neutral monosaccharides, 2-amino-2-deoxyhexoses and *N*-acetylhexosamines on untreated paper were ethyl acetate - acetic acid - water (9:2:2), or ethyl acetate - acetic acid - water - pyridine (9:2:2:1). Localisation was by spraying with 1% $KMnO_4$ soln. After washing off the excess of reagent wit water, brown spots indicated the sugar mercaptals. D. G. Moss

5145. **Modification of the spectrophotometric determination of ketone bodies in blood enabling the total recovery of β-hydroxybutyric acid.** J. Procos (Dept. of Biochem., Onderstepoort, S. Africa). *Clin. Chem.*, 1961, **7** (2), 97–106.—Acetoacetic acid in blood filtrate is converted into acetone by heating under reflux with H_2SO_4 in a micro-unit (Greenberg and Lester, *J. Biol. Chem.*, 1944, **154**, 177). For the determination of total ketone bodies, the blood filtrate is heated under reflux with H_2SO_4 to convert acetoacetic acid into acetone and the liquid is then heated under reflux again after addition of dichromate. This procedure ensures total recovery of the ketone bodies as acetone. The acetone is then determined by a modification of Thin and Robertson's method (*Biochem. J.*, 1952, **51**, 218) which increases the sensitivity; β-hydroxybutyric acid is obtained by difference. H. F. W. KIRKPATRICK

5146. **Quantitative enzymatic dehydrogenation of L(+)-lactate for micro-analysis.** G. Gercken (Inst. f. normal und pathol. Physiol., Univ., Köln, Germany). *Hoppe-Seyl Z.*, 1960, **320**, 180–186.—The use of an As_2O_3 - NaOH buffer (pH 9·6) in the presence of hydrazine sulphate enables the enzymatic determination of lactate by spectroscopic measurement of the reduced diphosphopyridine nucleotide formed to be carried out with none of the interfering reactions reported for other methods. The reaction is complete in 15 to 30 min. A rectilinear graph is obtained for the range 3·6 to 54 μmoles of lactate per ml. Recovery of added lactate is 99·1 ±3·1%. Lactate concn. in blood from the left ventricle and coronary sinus of the dog are about one-third of those found by other methods. F. POWELL

5147. **Improved method for determining pyruvic acid levels in tissues. Estimation of the aspartic-oxoglutaric and alanine-oxoglutaric transaminases.** F. Salvatore and V. Bocchini (Dept. of Biol. Chem., Univ. of Naples, Italy). *Clin. Chim. Acta*, 1961, **6** (1), 109–112 (in English).—Tissue homogenates (1 to 2%) are used. *Procedure*—To 0·5 ml of 0·2M-L-aspartic acid or L-alanine (pH 7·4) add 0·2 ml of 0·1M-2-oxoglutaric acid (pH 7·4) and dilute to 2 ml with phosphate buffer (pH 7·4). Place in a water bath at 37°, allow the temp. to reach that of the bath, then add 0·25 ml of the enzyme soln. Mix, and after exactly 10 min. add 0·2 ml of 50% trichloroacetic acid soln. Add 1 ml of 0·1% 2,4-dinitrophenylhydrazine soln. in 2·5N-HCl, allow the reaction to proceed for 5 min. at 37°, add 2 ml of xylene and shake vigorously for 5 min. Centrifuge at 3000 r.p.m. for 10 min., remove 1 ml of the xylene layer and add it to 6 ml of 2·5% KOH soln. in 95% ethanol. Measure the extinction at 490 mμ. Prepare standards by adding increasing quantities of Na pyruvate (0·4 to 2·0 μmoles) to aliquots of inactivated tissue homogenate containing 100 μmoles of amino-acid and decreasing quantities of 2-oxoglutaric acid (19·6 to 18·0 μmoles), keeping the total added keto-acids the same (20 μmoles). H. F. W. KIRKPATRICK

5148. **Determination of glyoxylic acid in biological systems.** B. A. McFadden and W. V. Howes (Dept. of Chem., State Univ., Pullman, Wash., U.S.A.). *Anal. Biochem.*, 1960, **1** (3), 240–248.—A potentiometric procedure is described for the determination of glyoxylate in amounts of 0·1 to 1 milli-equiv. and a colorimetric method for amounts of 0·025 to 0·03 μmole. *Potentiometric procedure*—The pH of an aliquot of the sample is adjusted to 9·5 with 0·5N-NaOH and 50 ml of M-Na_2SO_3 at the same pH is added. The liberated NaOH is titrated with 0·1N-H_2SO_4 to restore the pH to 9·5. *Colorimetric procedure*—An aliquot (5 ml) of the sample is treated with 1 ml of M-oxalic acid buffer (pH 1) and 1 ml of 1% phenylhydrazine hydrochloride soln. After boiling and cooling the mixture, 4 ml of conc. HCl and 1 ml of 5% $K_4Fe(CN)_6$ soln. are added and the extinction is measured at 520 mμ after 7 min. Glyoxal is the only substance likely to interfere seriously. J. R. HUDSON

5149. **Phosphoglycollic acid phosphatase [determination of phosphoglycollic acid].** K. E. Richardson and N. E. Tolbert (Dept. of Agric. Chem., Michigan State Univ., East Lansing, U.S.A.). *J. Biol. Chem.*, 1961, **236** (5), 1285–1290.—Three methods are described. Each depends on the cleavage of the acid into phosphoric acid and glycollic acid by the phosphatase. In the first method, the O consumed when the liberated glycollic acid is oxidised by glycollic acid oxidase is determined manometrically. In the second method, the free glycollic acid is determined colorimetrically by the standard method with 2,7-dihydroxynaphthalene. In the third method, the inorg. P is determined by one of the standard colorimetric methods. J. N. ASHLEY

5150. **Technique of paper-chromatographic separation and determination of the 2-oxo-acids in blood.** H. Käser (Univ.-Kinderklinik, Bern, Switzerland). *Clin. Chim. Acta*, 1961, **6** (3), 337–346 (in German).—The method of Cavallini *et al.* (*Nature*, 1949, **163**, 568; *Ibid.*, 1949, **164**, 792) is modified by use of new solvent systems, buffered papers and descending chromatography for a longer period. Separation and determination of 3-methyl-2-oxobutyric, 4-methyl-2-oxovaleric and 3-methyl-2-oxovaleric acids, in addition to 2-oxoglutaric and pyruvic acids, is possible by this means. H. F. W. KIRKPATRICK

5151. **Determination of 2-oxo-acids in body fluids and tissues.** E. Gründig (Med.-chem. Inst. d. Univ., Wien, Austria). *Clin. Chim. Acta*, 1961, **6** (3), 331–336 (in German).—A modification of the method of Friedman and Haugen (*J. Biol. Chem.*, 1943, **147**, 415) is described. H. F. W. KIRKPATRICK

5152. **Determination of urinary 4-hydroxy-3-methoxymandelic acid in man.** V. K. Weise, R. K. McDonald and E. H. Labrosse (Nat. Inst. of Mental Health, Bethesda, Md., U.S.A.). *Clin. Chim. Acta*, 1961, **6** (1), 79–86.—The method described combines ion-exchange separation of the 4-hydroxy-3-methoxymandelic acid from other phenolic acids with a radioactive-tracer technique. H. F. W. KIRKPATRICK

5153. **Determination of urinary 4-hydroxy-3-methoxymandelic acid by paper electrophoresis.** D. Klein and J. M. Chernaik (Long Island Lab. Inc., Flushing, N.Y., U.S.A.). *Clin. Chem.*, 1961, **7** (3), 257–264.—Dilute 3 ml of urine to 24 ml with H_2O, at the same time adjusting the pH to 2 with 3N-HCl. Place a 2-ml aliquot in each of three glass-stoppered tubes, heat in boiling H_2O for 10 min., cool and adjust the pH of one tube to 0·5 to 1·0 with 3N-HCl, discard, and add the same vol. of

acid to the other duplicate tubes. Extract with 4 ml, 2 ml and 2 ml of ethyl acetate, evaporate the combined extracts to dryness in a current of air at 40° to 50°, rinse the sides of the tubes with 0·5 ml of absolute ethanol and evaporate. Add ethanol dropwise to dissolve the residue and apply to a Whatman No. 3MM strip (3 cm × 30 cm): evaporate the solvent with a current of warm air as it is applied; also apply standard 4-hydroxy-3-methoxymandelic acid (**I**) (2·61 μg). Carry out the electrophoresis overnight in 0·5% (v/v) acetic acid at a constant current of 4 mA. Dry the strip at 80° for 30 min., then spray both sides with freshly prepared diazo reagent [mix 0·1% *p*-nitroaniline in 2% (v/v) HCl (10 ml), 0·2% $NaNO_2$ soln. (10 ml) and 10% K_2CO_3 soln. (20 ml), cooling in ice]. **I** appears as a purple band about half way toward the anode region of the paper. Cut out the **I** band whilst still moist, cut into pieces and place in a tube containing alkaline methanol [mix just before use 2% Na_2CO_3 soln. (25 ml) and absolute methanol (50 ml)]. Treat a piece of corresponding size from the unstained part of each strip similarly as blank. Stir occasionally during 1 hr., centrifuge and read the extinction at 520 mμ. Normal values found ranged from 1·1 to 3·9 μg of **I** per mg of creatinine, or an excretion of 1·2 to 3·4 μg per min. H. F. W. KIRKPATRICK

5154. **Effects of phenothiazine drugs on the determination of urinary 5-hydroxyindolylacetic acid.** B. T. Chadwick and J. H. Wilkinson (Westminster Med. Sch., London, England). *Clin. Chim. Acta*, 1960, **5** (6), 879–886.—The following method is recommended for use with patients on phenothiazine drugs. Collect a 24-hr. urine sample, with glacial acetic acid as preservative. Wash the urine (5 ml) with $CHCl_3$ (5 ml), acidify with 6N-H_2SO_4 (1 ml) and saturate with NaCl (approx. 2 g). Extract with ethyl ether (25 ml) for 15 min. and discard the aq. layer. Extract the ether with 0·5M-phosphate buffer soln. (pH 7·0) (5 ml) for 5 min. Discard the ether. To the buffer extract (4 ml) add 0·25% 1-nitroso-2-naphthol in ethanol (1 ml), mix and add HNO_2 reagent [mix 2N-H_2SO_4 (9 ml) and 5% aq. $NaNO_2$ (0·4 ml) immediately before use] (1 ml). Mix and incubate at 37° for 5 min., and extract with ethyl acetate (2 × 5 ml) for 5 min. to remove excess of reagent. Compare the colour with that given by an internal standard prepared by adding 5-hydroxyindolylacetic acid (100 μg) to the same urine (5 ml) and proceeding as described above. Prepare a reagent blank by treating phosphate buffer soln. (4 ml) with the colour reagents and extracting as described above. Read the extinction at 520 mμ.

H. F. W. KIRKPATRICK

5155. **Determination of quinic acid with special reference to its recovery from natural products.** J. Voigt and K. Rauscher (Inst. f. Ernährung, Dtsch. Akad. Wiss., Potsdam-Rehbrücke, Germany). *Nahrung*, 1961, **5** (3), 227–241.—A method is described in which the sample soln. (5 ml, containing 5 to 100 μg of quinic acid) is oxidised with 0·75% $NaIO_4$ soln. (1 ml) for 2 hr. at 22°; 1·0 ml of 0·5N-NaOH and 1·0 ml of 0·1M-alanine are added, and the mixture is made up to 10 ml and set aside for 4 hr. at 22°. The extinction is then measured at 381 mμ. The influence of various amino-acids, time and temp. of reaction and concn. of reagents is discussed. If interfering substances are present, the quinic acid must be separated by paper chromatography. I. DICKINSON

5156. **Procedure for isolation and determination of human blood angiotensin.** R. Boucher, P. Biron and J. Genest (Clin. Res. Dept., Hotel-Dieu Hosp., Montreal, Que., Canada). *Canad. J. Biochem. Physiol.*, 1961, **39** (3), 581–590.—Blood (200 to 400 ml) is added directly to 4 vol. of 95% ethanol, the amount of blood added being determined by wt. The alcohol extract is acidified with glacial acetic acid and concentrated to 100 ml and extracted with ethyl ether. The aq. layer is adjusted to pH 6 with aq. NH_3 and centrifuged; the supernatant fluid is added to a column of Dowex 50W-X2 (H^+). The column is eluted with 0·2N-ammonium acetate buffer (pH 6) and then with H_2O, these eluates being discarded. Angiotensin (**I**) is eluted with 0·1N-aq. NH_3, the eluate being neutralised at once with acetic acid and chromatographed on Whatman No. 2 paper. The isolated **I** is eluted from the strip and assayed by pressor activity in a rat preparation.

H. F. W. KIRKPATRICK

5157. **Photometric method for the determination of methylarbutin in bearberry leaves (Folia *Uvae ursi*).** B. Christ, G. Kühn and K. H. Müller (Arzneimittelwerk. A. Nattermann und Cie., Köln-Braunsfeld, Germany). *Arzneimittel-Forsch.*, 1961, **11** (2), 129–131.—Methylarbutin is hydrolysed with HCl to *p*-methoxyphenol, which is treated with $K_3Fe(CN)_6$ and 4-aminophenazone, and the product is determined photometrically at 455 mμ.

A. G. COOPER

5158. **Determination of bile pigments. II. Spectrophotometric determination of bilirubin and haemoglobin in serum.** N. Chiamori, R. J. Henry and O. J. Golub (Bio-Science Res. Foundation, Los Angeles, Calif., U.S.A.). *Clin. Chim. Acta*, 1961, **6** (1), 1–6.—Various spectrophotometric procedures are investigated and compared with the chemical method of Malloy and Evelyn (*J. Biol. Chem.*, 1937, **119**, 481). A good correlation between the results was found. It is also concluded that direct and indirect bilirubin have essentially the same absorption spectrum. Haemoglobin calculated from absorption formulae showed little correlation at low levels with results by the benzidine method. Icterus index determinations showed deviations from Beer's law with several light filters. H. F. W. KIRKPATRICK

5159. **Determination of bile pigments. III. Standardisation of the determination of urobilinogen as urobilinogen aldehyde.** R. J. Henry, S. L. Jacobs and S. Berkman (Bio-Science Res. Foundation, Los Angeles, Calif., U.S.A.). *Clin. Chem.*, 1961, **7** (3), 231–240.—Commercial prep. of urobilin and stercobilin were found to be too impure for use as standards. Phenolsulphonephthalein is proposed as an artificial standard, a 0·2 mg per 100 ml soln. of the acid form in 0·05% NaOH is equiv. to 0·346 mg per 100 ml of urobilinogen in the semi-quant. procedure of Watson *et al.* (*Amer. J. Clin. Path.*, 1944, **14**, 605), and to 0·307 mg per 100 ml in the quant. procedure (Watson, *Amer. J. Clin. Path.*, 1936, **6**, 458).

H. F. W. KIRKPATRICK

5160. **Estimation of direct and total bilirubin in serum. Investigations and observations by a modified method.** J. Brückner (Moncton Regional Lab., New Brunswick Dept. of Health and Social Services, Canada). *Clin. Chim. Acta*, 1961, **6** (3), 370–376.—The method described is a modification

of an earlier one (*Amer. J. Clin. Path.*, 1959, **32**, 513). The total bilirubin (**I**) is determined by diazotising and coupling with sulphanilic acid, with either phenazone or acetamide as accelerator. The direct-reading **I** is determined by diazotising for 5 or 30 min., stopping further reaction with phosphoric acid and coupling as before. Indirect **I** is given by difference. Haemolysis interferes with the determination when the concn. of **I** is elevated. H. F. W. KIRKPATRICK

5161. **Routine analysis of porphyrins in urine.** G. C. Mills (Dept. of Biochem. & Nutrition, Univ. of Texas Med. Branch, Galveston, U.S.A.). *Clin. Chem.*, 1961, **7** (2), 165–174.—A screening test is proposed in which the urine is diluted 1 to 5 with 0·5N-HCl and the extinction is read at 380 mμ; if the reading is $<0{\cdot}25$, the porphyrin concn. is below 0·3 μg per ml of urine. When the reading is $>0{\cdot}25$, purification may be carried out by means of Dowex 1-X8 anion-exchange resin (20 to 50 mesh, Cl^- form, no pre-treatment necessary). *Batch method*—Add the resin (1 g) to the urine (5 ml) in a test-tube, 2 cm diam., shake vigorously for about 30 sec. and allow the resin to settle. Pour off the supernatant fluid and discard, wash the resin with H_2O (2 × 20 ml) by mixing and decanting and give a third wash if the urine is highly coloured. Elute with 3N-HCl (2 × 10 ml) by mixing for 30 sec., allow to settle and pour off the eluate. Read the extinction of the mixed eluates at 380, 403, 406 and 430 mμ. *Column method*—Prepare a column (6 cm × 1 cm) of the resin with H_2O, restricting the flow rate to $\geqslant 4$ ml per min. Add the urine (5 ml) to the column and allow to drip through, wash with H_2O (20 ml), giving a further wash if necessary to remove colour. Elute with 3N-HCl (2 × 15 ml) and determine the extinction as before. A correction equation is applied, $P_{max.} = [2D_{max.} - (D_{380} + D_{430})]/1{\cdot}84$, where $P_{max.}$ is the max. absorption due to porphyrins and $D_{max.}$ is the observed max. absorption, and the wavelength of which varies with the predominating porphyrin; $P_{max.}/0{\cdot}580 =$ μg of porphyrins per ml in the 3N-HCl soln. as uroporphyrin. H. F. W. KIRKPATRICK

5162. **Chromatography of porphyrins and metalloporphyrins.** J. E. Falk (C.S.I.R.O. Div. of Plant Industry, Canberra, Australia). *J. Chromatography*, 1961, **5** (4), 277–299 (in English).—A review, with 91 references, is presented. In paper chromatography, the value of lutidine - H_2O mixtures as solvents is stressed. Column chromatography on magnesia, alumina and calcium carbonate is described. There is some account of the chromatography of haems, and electrophoresis is also briefly mentioned. Data are tabulated for spectrophotometric or fluorimetric determinations on separated spots. G. RUSSELL

5163. **Plasma porphyrin.** F. S. Schlenker, C. L. Davis and C. L. Kitchell (Biochem. Res. Sect., Med. Res. Lab., Veterans Admin. Med. Teaching Group Hospital, Memphis, Tenn., U.S.A.). *Amer. J. Clin. Path.*, 1961, **36** (1), 31–36.—Plasma, diluted with an equal vol. of water and adjusted to pH 1·0 with HCl, is passed through a column of Florisil (60 to 100 mesh) which has been preheated during manufacture to 260°. After washing the column with water, the porphyrin is eluted with acetone - ether - glacial acetic acid (5:5:1) and then extracted from the eluate with 2·5N-HCl in 10% NH_4Cl soln. The porphyrin content is calculated from the extinction of the relatively pure acid porphyrin soln. at various wavelengths between 380 and 430 mμ, the equations devised by Sveinsson, Rimington and With being used in the calculation. R. A. BRENAN

5164. **Quantitative separation and estimation of glycosphingosides in nervous tissue with special reference to gangliosides.** S. N. Payne and B. S. Platt (Nat. Inst. for Med. Res., Mill Hill, London, N.W.7). *J. Neurochem.*, 1961, **7** (1), 5–15.—Ethanolic extracts of nervous tissue (dog and rat brain) obtained by a specified procedure were dissolved in $CHCl_3$ - methanol (10:1) and separated on a column of silicic acid (300 mesh, activated by heating at 135° for 6 hr.) by gradient elution with $CHCl_3$ - methanol mixtures (Weiss, *J. Biol. Chem.*, 1956, **223**, 523); 17 fractions were obtained and determined by evaporation and weighing. Those comprising individual peaks were pooled, giving 6 main fractions. Fractions (*a*) to (*c*) were examined for hexoses by hydrolysis with 2N-HCl followed by paper chromatography, and fractions (*d*) to (*f*) for *N*-acetylneuraminic acid by Bial's reaction (Werner and Odin, *Acta Soc. Med. Upposala*, 1952, **57**, 230), for hexosamines (Svennerholm, *Ibid.*, 1956, **61**, 287; Gardell, *Acta Chem. Scand.*, 1953, **7**, 207) and for hexoses. Hydrolysates of all fractions were examined qualitatively for amino-acids by paper chromatography. The results are discussed. Fraction (*b*) appears to be kerasin, (*c*), phrenosin, (*d*) and (*e*), gangliosides and (*f*), "carnithin". It appears that fraction (*d*) is not a cerebroside and not a true ganglioside; it may be an intermediate product. A high result for hexosamine, due to the presence of "carnithin," is obtained on $CHCl_3$ - methanol extracts of nervous tissue, and it is suggested that for the accurate determination of ganglioside both *N*-acetylneuraminic acid and hexosamines should be determined. R. E. E.

5165. **New micro-method for the estimation of serum total fatty acids.** J. Drysdale and J. D. Billimoria (Westminster Med. Sch., London, England). *Clin. Chim. Acta*, 1960, **5** (6), 828–833.—The serum is extracted with ethanol - ethyl ether (3:1), and an aliquot of the extract is heated with NaOH soln. to hydrolyse the esters. After neutralising with H_2SO_4 the fatty acids are extracted into heptane and titrated with standard NaOH soln. to thymol blue. Recovery and results for normal subjects are given. H. F. W. KIRKPATRICK

5166. **Micro-determination of polyenoic fatty acids and total fatty acids in plasma and tissue.** J. G. Nørby (Biokem. Inst., Univ., Århus, Denmark). *Acta Chem. Scand.*, 1961, **15** (3), 525–534 (in English).—The total fatty acids are extracted from 2 g of sample with ethanol - ether (3:1). After saponification with ethanolic KOH, the ethanol content is adjusted to 50% (v/v) and the unsaponifiable fraction is extracted with light petroleum (boiling-range 30° to 45°). The total cholesterol may be determined on an aliquot. The fatty acids are liberated from an aliquot of the ethanolic soln. with 4N-HCl and extracted in the same way. They are then titrated with 0·025N-NaOH. The polyenoic fatty acids are determined by the method of Herb and Riemenschneider (*Anal. Chem.*, 1953, **25**, 953). The saponification method and its effect on changes in composition and structure of the fatty acids were investigated fully and results show the method to be preferable

to that of Pikaar and Nijhof (*Anal. Abstr.*, 1959, **6**, 669). Pyruvic, lactic, β-hydroxybutyric and citric acids do not interfere. J. Hall

5167. **A new method for the detection of diglycerides on a micro scale.** B. F. C. Clark (Univ. Chem. Lab., Lensfield Road, Cambridge, England). *J. Chromatography*, 1961, **5** (4), 368–369 (in English).—Diglycerides (**I**) may be distinguished from monoglycerides (**II**) on paper chromatograms by the fact that **II** react directly with periodate - Schiff's reagent, whereas **I** require prior decomposition, *e.g.*, with hydroxylamine. *Procedure*—Spot the mixture of **I** and **II** (20 μg in 20 μl) on Whatman No. 1 paper impregnated with formaldehyde (Hörhammer *et al.*, *Anal. Abstr.*, 1960, **7**, 5361). Develop for 12 hr. (≃25 cm) by the ascending method with n-butanol - acetic acid - water (4:1:5). Dry and divide the paper lengthwise into two so as to obtain spots of **I** and **II** on each strip. Spray one strip with hydroxylamine soln. (equal vol. of 2·5M-NaOH and 2M-hydroxyammonium chloride). After 30 min. spray with 3M-HCl, dip in H_2O for 30 sec. and allow to dry. Spray both strips with aq. $NaIO_4$ soln. (1%) and, after 6 min., treat with SO_2 gas to bleach free iodine. Spray at once with Schiff's reagent (1%) and again treat with SO_2 gas. The detection limit for the mauve spots due to **I** and **II** is 5 to 10 μg. R_F values are quoted, and the method is applied to the determination of the structure of horse-liver monophospho-inositide. G. Russell

5168. **Suggested modifications of the micro-determination of triglycerides.** E. Van Handel and R. Ordway (Entomol. Res. Centre, P.O. Box 308, Vero Beach, Fla., U.S.A.). *Clin. Chem.*, 1961, **7** (3), 249–251.—Modifications of the method of Van Handel and Zilversmit (*Anal. Abstr.*, 1958, **5**, 647) are described. The triglycerides in this method are measured by glycerol assay. H. F. W. Kirkpatrick

5169. **Simplified determination of the lipid components of blood serum.** H. V. Connerty, A. R. Briggs and E. H. Eaton, jun. (Vet. Admin. Hosp., Wilmington, Del., U.S.A.). *Clin. Chem.*, 1961, **7** (1), 37–53.—Methods are described for the determination of total fatty acids as their esters by the hydroxamic acid reaction, phospholipids by copptn. with protein and determination of P in the ppt., and free and total cholesterol by a simplified procedure in which ferric iron reagent is used. H. F. W. Kirkpatrick

5170. **Chromatography of lipids on silicic acid; infra-red spectrophotometric elution curves.** J. J. Wren and P. M. Lenthen (Lyons Lab., Hammersmith Road, London, W.6). *J. Chromatography*, 1961, **5** (4), 370–371 (in English).—The lipids were separated with a concave gradient of methanol in $CHCl_3$ and the fractions measured at 1745 cm^{-1} (stretching frequency of C=O in esters). Results compared well with gravimetric elution curves. G. Russell

5171. **Thin-layer chromatography of brain lipids, their derivatives and breakdown products.** H. Jatzkewitz and E. Mehl (Dtsch. Forschanst. f. Psychiatrie, München, Germany). *Hoppe-Seyl Z.*, 1960, **320**, 251–257.—Chromatography on a thin layer of silicic acid is very suitable for the examination of brain lipids, for which it is of general application. It is less sensitive to temp., quicker and gives sharper zones than paper. R_F values are given of 30 lipids when examined by thin-layer chromatography in 11 solvent systems. For surveying the components of unknown lipid mixtures, 3 solvent systems in succession are recommended. Weakly alkaline bromothymol blue soln. is recommended as a general reagent for detection (sensitivity 0·1 to 1·0 μg). Special reagents for the detection of cholesterol, monoglycerides and phospho- and glyco-lipids are described. F. Powell

5172. **Detection of coeruloplasmin after zone electrophoresis.** J. A. Owen and H. Smith (St. Vincent's Hosp., Melbourne, Australia). *Clin. Chim. Acta*, 1961, **6** (3), 441–444.—The stain described previously for the detection of haemoglobin and haemoglobin - haptoglobin complexes (**I**) (Owen *et al.*, *Anal. Abstr.*, 1959, **6**, 3699) has been modified, thus increasing sensitivity. This has led to the observation of a reactive zone after starch-gel electrophoresis of normal serum whether **I** is present or not, subsequently identified as coeruloplasmin (**II**). The staining reagent for **II** is made by mixing 1% *o*-dianisidine soln. (in H_2O, with just sufficient HCl for complete dissolution) (10 ml), acetate buffer (pH 5·7) (10 ml), 95% ethanol (30 ml), and H_2O to 100 ml. After electrophoresis, at pH 6·5 for best separation, the strip is placed in the reagent for 2 hr. at 37° and is then washed with H_2O. **II** gives a pink band on paper and an orange - red band on starch gel. The colour is stable for at least 6 months. H. F. W. Kirkpatrick

5173. **Separation of human haemoglobins by starch-gel zone electrophoresis.** R. L. Engle, jun., A. Markey, J. H. Pert and K. R. Woods (Dept. of Med., New York Hosp., Cornell Med. Centre, N.Y., U.S.A.). *Clin. Chim. Acta*, 1961, **6** (1), 136–141.—Excellent resolution of normal and abnormal haemoglobins is obtained on this medium with relatively little trailing of components. Separation occurs of haemoglobin A_2, two other pigmented fractions and two non-pigmented protein fractions in normal red cell haemolysates, and the method can be used to distinguish between foetal haemoglobin and haemoglobin A when both are present. H. F. W. Kirkpatrick

5174. **A comparison of three methods for the assay of haemoglobin.** A. Kumlien, K. Paul and S. Ljungberg (Biokem. Inst., Stockholm, Sweden). *Scand. J. Clin. Lab. Invest.*, 1960, **12** (4), 381–383.—The haemoglobin content of three samples of blood has been determined as cyanomethaemoglobin, oxyhaemoglobin, and pyridine haemochromogen. The last method is believed to give the correct values, but the differences between the results are small enough to be negligible in routine work. P. Pedersen

5175. **Determination of carboxyhaemoglobin.** T. P. Whitehead and S. Worthington (Grp. Path. Lab., Warwick, England). *Clin. Chim. Acta*, 1961, **6** (3), 356–359.—The method of Wolff (*Ann. Méd. Lég.*, 1947, 221) is modified to give accuracy especially at lower concn. *Procedure*—Mix the blood (1 ml) with normal saline (9 ml) in a centrifuge tube and centrifuge for 5 min. Discard the clear supernatant liquid and repeat the washing with further saline (9 ml). Add H_2O (4·5 ml) to the cells, mix well and place 1 ml (*T*) in a 6 in. × 0·5 in. test-tube. Place the remainder in a 100-ml conical

flask and bubble coal gas through it for 1 min., shaking vigorously. Place 1 ml of this soln. (*C*) in another test-tube. To both tubes add 40·8% (w/v) Na acetate (cryst.) soln. (3 ml) and 28% (v/v) acetic acid (0·75 ml), mixing after each addition. Heat the tubes in a water bath at 57° to 57·5° for exactly 8 min., cool and filter through 7-cm No. 1 Whatman papers. Read the extinction at 555 mμ, diluting *C* 1 in 10, and *T*, if necessary, to bring to approx. the same reading. The carboxyhaemoglobin (%) is given by (reading of *T* × dilution/reading of *C* × dilution) × 100.

H. F. W. KIRKPATRICK

5176. **The determination of thiamine in urine by the thiochrome method.** H. Haugen (Rikshospitalet, Oslo, Norway). *Scand. J. Clin. Lab. Invest.*, 1960, **12** (4), 384–391.—In the method described, benzenesulphonyl chloride is used as a blocking reagent for the determination of the blank. The method is simple and more rapid than previous procedures. P. PEDERSEN

5177. **Excretion of 4-aminoimidazole-5-carboxamide in human urine.** P. L. McGeer, E. G. McGeer and M. C. Griffin (Kinsmen Lab. of Neurol. Res., Univ. of British Columbia, Vancouver, Canada). *Canad. J. Biochem. Physiol.*, 1961, **39** (3), 591–603.—This compound is a product of purine biosynthesis and may be an indicator of the state of this particular process in humans under normal and pathological conditions. *Procedure*—Acidify 125 ml of salt-saturated urine to pH 2 and extract with ethyl acetate (5 × 40 ml). Make 50 ml of the aq. soln. alkaline with aq. NH_3 - pyridine (1:4) and extract with n-butanol - pyridine (20:1) (5 × 26 ml). Acidify the combined organic extracts with conc. H_2SO_4 added dropwise and separate the aq. layer that forms. Wash the butanol layer with small portions of 0·2N-H_2SO_4 (× 5) and combine the aq. soln. (20 to 25 ml total vol.). To an aliquot (2 ml) add acetic anhydride (2 drops) with vigorous shaking and allow to stand for 20 min. Add 0·2N-H_2SO_4 (3 ml) and 0·1% $NaNO_2$ soln. (0·5 ml); exactly 2 min. later add 0·5% ammonium sulphamate soln. (0·5 ml) followed immediately by 0·1% *N*-1-naphthylethylenediamine soln. (0·5 ml). Read the extinction at 540 mμ after 20 min. agai st a blank.

H. F. W. KIRKPATRICK

5178. **Determination of uric acid in biological fluids. I. Modification of the method of Bergmann and Dikstein.** M. L. Wharton and D. R. A. Wharton (Pioneering Res. Div., Quartermaster Res. and Engng Center, Natick, Mass., U.S.A.). *Anal. Biochem.*, 1960, **1** (3), 213–217.—The method of Bergmann and Dikstein (*Anal. Abstr.*, 1955, **2**, 717), based upon pptn. of uric acid with mercuric acetate followed by dissolution in NaCl soln. and measurement of the extinction at 290 mμ, has been modified to deal with very low concn. (≃ 4 μg per ml). Recoveries of 5 μg of uric acid per ml were ≃ 95%. For urine samples that contained up to 1300 μg of uric acid per ml, results by the new method agreed well with those by the original method and by the method of Kalckar (*J. Biol. Chem.*, 1947, **167**, 429). J. R. HUDSON

5179. **Analysis of purines and pyrimidines by elution from columns of sulphonated polystyrene with buffers of pH 4.** C. F. Crampton, F. R. Frankel, A. M. Benson and A. Wade (Dept. of Pathol. and Biochem., Coll. Med. Univ. Florida, Gainsville, U.S.A.). *Anal. Biochem.*, 1960, **1** (3), 249–262.—Detailed procedures are given for the separation of uracil, thymine, guanine, cytosine, adenine and 5-methylcytosine on columns of Dowex 50-X2 or -X4 with Na citrate or ammonium formate buffers of gradually increasing ionic strength as eluents. The u.v. absorption characteristics of soln. of the compounds named in the buffers are tabulated, and elution curves are given.

J. R. HUDSON

5180. **Analysis for guanidine and methylguanidine in uraemic plasma.** M. H. Carr and P. R. Schloerb (Res. Lab., Veterans Admin. Hosp., Kansas City, Mo., U.S.A.). *Anal. Biochem.*, 1960, **1** (3), 221–227.—After removal of protein and arginine, by treatment with $Ba(OH)_2$ and $ZnSO_4$ and filtration, methylguanidine (**I**) and guanidine (**II**) are separated from plasma by adsorption on a column (1 cm × 6 mm) of strong cation-exchange resin (Dowex 50W-X8, 100 to 200 mesh). The column is washed with 0·1M-NaCl and eluted with 4N-KOH, and aliquots are used to determine **I** by the Sakaguchi procedure. Further aliquots are treated with dimethyl sulphate, to convert **II** into **I** before colorimetry. The standard deviations are 0·004 mg per 100 ml for **I** and 0·005 mg per 100 ml for **II**.

J. R. HUDSON

5181. **Micro-determination of amino-nitrogen as copper complexes. A modification for plasma and urine.** M. Kekki (Wihuri Res. Inst., Helsinki, Finland). *Scand. J. Clin. Lab. Invest.*, 1959, **11** (4), 311–321.—A new micro-method is described for the indirect determination of the amino-N content of plasma and urine, in which the Cu content of the soluble Cu - amino-acid complex is determined as the coloured complex formed with biscyclohexanone oxalyldihydrazone (**I**) or with oxalyldihydrazide (**II**). *Prodedure for urine*—Filtered diluted urine (1:59) (3 ml) is treated with copper phosphate suspension (2 ml) (prep. described). After 30 min. the ppt. is removed by centrifuging. Water is used as a blank. Copper is determined spectrophotometrically with either **I** or **II**. *Procedure for plasma or serum*—To plasma or serum (0·1 ml) add H_2O (1 ml) and 0·004N-H_2SO_4 (0·7 ml). This mixture is heated at 95° to 98° for 2 min. The protein is pptd. by adding 0·004N-H_2SO_4 (0·6 ml for plasma or 0·5 ml for serum), and the heating is then continued for 8 min. The tubes are cooled, and the vol. is brought to 3 ml. After centrifuging, the supernatant liquid (1·5 ml) is treated with 1·0 ml of the copper phosphate suspension, and the Cu is determined as for urine except that two blanks, water and 0·004N-H_2SO_4 treated with the copper phosphate suspension, are used. Reagent **II** is preferred because of its greater stability and sensitivity.

D. B. PALMER

5182. **Quantitative determination of amino-acids by combined displacement and paper chromatography.** F. Lundquist and H. Galatius-Jensen (Univ. Inst. of Forensic Medicine, Copenhagen, Denmark). *Scand. J. Clin. Lab. Invest.*, 1960, **12** (3), 342–352.—A fractionation of all the common amino-acids into 5 groups is performed on an ion-exchange column by carrier displacement chromatography. An arrangement is described which permits 4 analyses to be carried out at the same time. The amino-acids in each of the groups are separated by one- or two-dimensional paper

chromatography, and the determination is performed on the spots after preliminary ninhydrin staining, followed by extraction. Separate measurements, such as colorimetric analysis for proline, hydroxyproline and histidine, may be performed directly on the fractions. The method permits the determination of all the common amino-acids in a mixture containing 1 to 10 μmoles of each. The accuracy for most amino-acids is similar to that of Moore and Stein's method. P. PEDERSEN

5183. **Semi-micro method for the determination of amino-acids by ion-exchange chromatography.** J. E. Eastoe (Dept. of Dental Sci., Roy. Coll. of Surg. of England, London). *Biochem. J.*, 1961, **79** (3), 652–656.—This modification of the Moore and Stein ion-exchange method (*Brit. Abstr. C*, 1949, 234; 1952, 62) as modified by Eastoe (*Anal. Abstr.*, 1956, **3**, 1461) allows a complete analysis to be carried out on 0·3 to 0·6 mg of protein. The sensitivity of the method is increased owing to a decrease in the diameter of the resin column from 0·9 to 0·4 cm with a corresponding reduction of the original volume of the fractions and reagents by 80%. The preliminary separation of basic amino-acids is effected on a column (2·2 cm × 0·4 cm) of Dowex 50 at pH 5·0, and hydroxylysine is separated from histidine by combining the small column with a 15-cm column and eluting the two columns in series. The sharpness and resolution of the amino-acid peaks are unaffected by the decrease in diameter of the column. The over-all accuracy of the method is ≃5%. Colorimetry on the reduced scale is very similar to that in the original method, but the rate of fading is increased. A simple method is described for modification of a spectrophotometer so that convenient measurements can be made on small vol. of soln. in standard optical cells. J. N. ASHLEY

5184. **Modified procedure for the automatic analysis of amino-acids.** K. A. Piez and L. Morris (Nat. Inst. of Dental Res., U.S. Dept. of Health, Educ. and Welfare, Bethesda, Md.). *Anal. Biochem.*, 1960, **1** (3), 187–201.—A detailed description is given of the apparatus and reagents for, and the method of operation of, an automatic system developed from that of Spackman *et al.* (*Anal. Abstr.*, 1958, **5**, 4359). A single column of Dowex 50-X12 resin (highly cross-linked) and a Varigrad gradient-producing device (Peterson and Sober, *Anal. Abstr.*, 1959, **6**, 5025) are used, and the analysis of a protein hydrolysate can be carried out in 24 hr. with ⩾2 hr. of operator time. Regeneration of the column is achieved during the analysis, since gradient elution completely removes the amino-acids. J. R. HUDSON

5185. **Gas-liquid chromatography of amino-acid derivatives.** D. E. Johnson, S. J. Scott and A. Meister (Dept. of Biochem., Tufts Univ. Sch. of Med., Boston, Mass., U.S.A.). *Anal. Chem.*, 1961, **33** (6), 669–673.—A standard procedure is described for the preparation of the derivatives, and columns are described for the separation of 35 amino-acids, including 18 of the common protein amino-acids. Preparation of the derivatives and chromatography, on 2 to 8-ft. columns packed with Chromosorb W coated with 0·5 to 5% of polyoxyethylene glycol 1540 or 6000, at 125° to 155°, takes about 2 hr. and amounts of amino-acid of the order of 10^{-10} mole can be detected by means of strontium-90 ionisation detectors. The method is applicable to the analysis of hydrolysates of small peptides eluted from paper chromatograms, to the detection of impurities in commercial samples of amino-acids and to the identification of minute amounts of amino-acids present in preparations of soluble ribonucleic acids. C. B. BAINES

5186. **Specific method for the analysis of hydroxyproline in tissues and urine.** D. J. Prockop and S. Udenfriend (National Heart Inst., U.S. Dept. of Health, Educ. and Welfare, Bethesda, Md., U.S.A.). *Anal. Biochem.*, 1960, **1** (3), 228–239.—Procedures for the analysis of (*i*) relatively pure soln. of hydroxyproline and (*ii*) samples of tissue, protein and urine are described. In (*i*) a sample containing 0·01 to 0·1 μmole of hydroxyproline is treated with phosphate - borate buffer (pH 8·7) and oxidised with chloramine-T soln. to give pyrrole, which is then extracted with toluene and determined colorimetrically with *p*-dimethylaminobenzaldehyde at 560 mμ. In (*ii*) the sample is hydrolysed with HCl, and pigments are removed with a mixture of cation-exchange resin and activated charcoal before the oxidation step, which is carried out in the presence of a measured excess of alanine. Recoveries of hydroxyproline from casein hydrolysates, urine and liver tissue were usually better than 87%. J. R. HUDSON

5187. **Estimation of phenylalanine in serum by paper chromatography at elevated temperatures.** J. M. Van de Heuvel and S. K. Wadman (Municipal Hosp., Zuiderziekenhuis, Rotterdam, The Netherlands). *Clin. Chim. Acta*, 1960, **5** (6), 824–827 (in English).—In the method described, the serum is deproteinised with ethanol and the alcoholic extract is passed through a column of Dowex 50W-12 (50 to 100 mesh). The adsorbed amino-acids are eluted with 3·5% aq. NH_3 and the eluate is evaporated to dryness. The residue is dissolved in H_2O (0·1 ml) and chromatographed on Whatman No. 1 paper with benzyl alcohol saturated with H_2O, the chromatography being carried out for 15 hr. at 37°. The phenylalanine is located and determined with ninhydrin. H. F. W. KIRKPATRICK

5188. **The determination of free urinary tyrosine.** M. Duval and J. Delga (Lab. de Chim. et Inst. Prophylactique, Val-de-Grâce, France). *Ann. Pharm. Franç.*, 1961, **19** (2), 94–103.—The optimum conditions for the determination of tyrosine by the methods of Folin and Ciocalteu (*J. Biol. Chem.*, 1927, **73**, 627) and Udenfriend and Cooper (*J. Biol. Chem.*, 1952, **196**, 227) are investigated. Neither method can be applied directly to urine, and pretreatment by the following method is necessary. The urine is acidified to pH 3 to 4 and 15 ml of it is allowed to percolate through a column of 10 g of Amberlite IR-120 resin at 1 ml per 4 min. The bed is washed with 300 ml of water and the amino-acids are then eluted with 40 ml of aq. NH_3 (diluted 1:2) at 1 ml per 3 min.; the eluate is evaporated to dryness and the residue is dissolved in 3 ml of 2N-H_2SO_4 for Folin's method, or the eluate is treated directly by Udenfriend's method. In testing urine, a high result is obtained by Folin's method with urine from patients who have received aspirin, unless the urine is extracted with ethyl ether. E. J. H. BIRCH

5189. **A note on the determination of tryptophan.** D. F. Evered (Chelsea Coll. of Sci. and Technol., London, S.W.3). *Anal. Biochem.*, 1961, **2** (3), 280–281.—Attempts to use Searcy's method (*Anal.*

Abstr., 1959, **6**, 3693) for the determination of free tryptophan in deproteinised blood plasma gave very low colour yields. *N*-Acetyl- and *N*-benzoyl-tryptophan gave much stronger colours than free tryptophan. Blocking the α-amino-group of tryptophan by acylation or by peptide-link formation, as in a protein, is thought to prevent loss of tryptophan by some side reaction during the analysis. D. G. Moss

5190. **Spectrophotometric determination of homocysteine thiolactone.** Masao Maruyama and Yasuo Morotomi (Sankyo Co., Ltd., Shinagawa-ku, Tokyo). *Ann. Rep. Takamine Lab.*, 1959, **11**, 118–121.—Homocysteine thiolactone was hydrolysed at pH >13 under N to homocysteine. After neutralisation, the soln. was treated with *N*-ethylmaleimide in phosphate buffer (0·1M, pH 6·0). The decrease in the extinction at 300 mμ of the reaction mixture was measured and the concn. of homocysteine was calculated. S. Natori

5191. **Method for the determination of taurine in urine.** B. Sörbo (Res. Inst. of Nat. Defence, Dept. 1, Sundbyberg, Sweden). *Clin. Chim. Acta*, 1961, **6** (1), 87–90 (in English).—Dowex 50W-X8 (100 to 200 mesh) is used in the H^+ form as a 0·8 cm × 5 cm column. Apply 1 ml of protein-free urine to the top of the column and wash the resin with H_2O (3 × 2 ml) keeping the flow rate < 1 ml per min. To the total percolate add methyl red (0·02% in 95% ethanol) (1 drop) and titrate with 0·2M-NaOH, then dilute to 10 ml. To 1 ml add 2 ml of ninhydrin reagent, cap the tube and heat in boiling H_2O for 20 min., cool, dilute with 50% ethanol (5 ml) and read at 570 mμ in a 1-cm cell. A blank is prepared with H_2O instead of percolate. A standard curve for taurine is linear up to 5 μmoles per ml of urine. If the urine contains protein, adjust 5 ml to pH 6 to 8, dilute to 6·5 ml, add 10% $ZnSO_4.7H_2O$ soln. (1 ml) and 0·2M-NaOH (2 ml), centrifuge and analyse 2 ml of supernatant fluid by the above process. Prepare the ninhydrin reagent by mixing 3% ninhydrin soln. in redistilled peroxide-free 2-methoxyethanol (25 ml), 0·2M-citrate buffer (pH 5·70) (25 ml) and $SnCl_2$ soln. (4·51 g of $SnCl_2.2H_2O$ dissolved in 1·7 ml of conc. HCl and diluted to 10 ml with H_2O) (0·10 ml).
H. F. W. Kirkpatrick

5192. **Method for the determination of free, non-protein-bound thyroxine in serum.** L. K. Christensen (Gentofte Hosp., Copenhagen, Denmark). *Scand. J. Clin. Lab. Invest.*, 1959, **11** (4), 326–331.—A tracer dose of ^{131}I-labelled thyroxine (**I**) in buffer soln. is added to serum and the mixture is incubated at 37° for 90 min. to establish equilibrium with respect to protein binding. This soln. is then dialysed in a special apparatus, details of which are given, against buffered serum without added **I**. The % of the total amount of added **I** passing through the membrane in 24 hr. gives a measure of the free **I** present, and if this value is multiplied by the serum protein-bound iodine, determined separately, an expression of the free thyroxine present in the serum is obtained in arbitrary units, since the proposed method does not give an absolute value. Reproducibility of the results is critically dependent upon using the same manufacture of membrane, and apparatus of exactly similarly dimensions. It is also essential that the **I** used be free from impurities. Results with sera from normal individuals showed that 0·46 to 0·66% of the total **I** added passed through the membrane in 24 hr. H. F. W. Kirkpatrick

5193. **Chromatography of aromatic nitro-derivatives on polyamide.** W. Grassmann, H. Hörmann and H. von Portatius (Max-Planck Inst. f. Eiweiss- und Leder-forsch., München, Germany). *Hoppe-Seyl Z.*, 1960, **321**, 120–132.—The behaviour and separations of various 2,4-dinitrophenyl derivatives of amino-acids and peptides and certain aromatic nitro-compounds when chromatographed on polycaprolactam have been investigated in solvent systems containing dimethylformamide, acetic acid, water and ethanol. The results, and the fact that acetylated polycaprolactam has very little affinity for nitro-compounds, show that interaction takes place between the nitro-group and the free amino-groups of the polymer. Phenols, which behave similarly on both untreated and acetylated polyamide, are considered to be bound by the peptide bonds. F. Powell

5194. **Dye-binding by protein as applied to quantitative paper electrophoresis.** D. A. Osborn (Inst. of Laryngology and Otology, Univ. of London, England). *Clin. Chim. Acta*, 1960, **5** (6), 777–794.—The uptake of dye is investigated with special reference to Light green SF (C.I. Acid Green 5). It is shown to vary with the method of denaturation, the nature of the dye solvent, and with the temp. and duration of washing. Even at low concn. of protein the uptake is not in linear relationship with concn. Comparison is made with Lissamine green SF (C.I. Acid Green 7) and bromocresol green. H. F. W. Kirkpatrick

5195. **Determination of protein by the biuret reaction using cupric hydroxide suspension as reagent.** P. Siltanen and M. Kekki (Wihuri Research Inst., Helsinki, Finland). *Scand. J. Clin. Lab. Invest.*, 1960, **12** (2), 228–234.—Applications of a modified biuret method to biological analysis are discussed. A specially prepared suspension of $Cu(OH)_2$ in KOH soln. is used. P. Pedersen

5196. **Value and reliability of the copper-Folin method for the determination of very small amounts of protein.** H. P. Rieder (Neurolog. Res. Lab., Univ.-Poliklinik, Basle, Switzerland). *Clin. Chim. Acta*, 1961, **6** (2), 188–194 (in German).—This method gives very reliable results for the determination of protein in c.s.f. except when the patient is receiving certain drugs, particularly salicylic acid and 4-aminosalicylic acid. These are detected by their u.v. absorption.
H. F. W. Kirkpatrick

5197. **Estimation of serum or plasma protein using qualitative Benedict reagent.** Q. Z. Hussain, N. S. Shah and S. N. Chaudhuri (Maulana Azad Med. Coll., New Delhi, India). *Clin. Chim. Acta*, 1961, **6** (3), 447–448.—Dilute the plasma or serum 1 to 5 with normal saline and Benedict's qualitative reagent (1 to 10) with H_2O. Mix the dil. serum (1 ml) with dil. Benedict's reagent (4 ml) and add 30% NaOH soln. (0·5 ml). Read the extinction at 555 mμ after 30 min. Fractionated proteins may be determined similarly.
H. F. W. Kirkpatrick

5198. **Micro-determination of protein by determining the protein-bound copper of the biuret complex with oxalyldihydrazide.** M. Kekki and P. Siltanen (Wihuri Research Inst., Helsinki,

Finland). *Scand. J. Clin. Lab. Invest.*, 1960, **12** (2), 235–238.—A micro-method for protein determination is presented, in which the protein-bound copper in a modified biuret method (*cf.* Siltanen and Kekki, *Anal. Abstr.*, 1961, **8**, 5195) is estimated colorimetrically with oxalyldihydrazide instead of by the biuret colour itself. The method can be used over the range 0·01 to 0·75 mg of protein.
P. PEDERSEN

5199. **Electrophoretic separation of proteins on emulsions of adsorbents.** W. Ostrowski (Dept. of Physiol. Chem., Acad. of Med., Krakow, Poland). *Clin. Chim. Acta*, 1961, **6** (1), 38–43 (in English).—Various adsorbents, *e.g.*, starch, cellulose powder, Celite 535, titanium dioxide and Hyflo-Supercel, prepared as emulsions in 0·2% agar dissolved in a suitable buffer soln., were tested as media for the electrophoretic separation of proteins. Best results were given by Hyflo-Supercel in concn. of 1 to 5%.
H. F. W. KIRKPATRICK

5200. **Rapid estimation of albumin and total protein in small amounts of blood serum.** J. W. Keyser (Royal Infirmary, Cardiff, Wales). *Clin. Chim. Acta*, 1961, **6** (3), 445–447.—The methyl orange method of Bracken and Klotz (*Amer. J. Clin. Path.*, 1953, **23**, 1035) for albumin is used with the inclusion of a serum blank to eliminate the effect of turbidity, icterus and slight haemolysis. Total protein is estimated on the serum blank, after determination of albumin, by incubation with a biuret reagent. H. F. W. KIRKPATRICK

5201. **Adsorption of serum mucoproteins on filter-paper.** M. Ledvina and S. Coufalova (Central Biochem. Lab., Gottwaldov Hosp., Czechoslovakia). *Clin. Chim. Acta*, 1961, **6** (1), 16–21 (in German).—All types of paper were found to adsorb mucoproteins, the amount varying from 6·5 to 19·3%. Lowest adsorption was found with Whatman No. 4 filter-paper. This adsorption constitutes a source of error in the determination of these compounds.
H. F. W. KIRKPATRICK

5202. **Improved rapid procedure for detection of high protein-bound iodine levels.** H. Strickler, Y. Cannon, E. Campbell and J. Shellito (William H. Singer Mem. Res. Lab., Allegheny Gen. Hosp., Pittsburgh, Pa., U.S.A.). *Clin. Chem.*, 1961, **7** (3), 294–296.—The method of Grossman (*Ibid.*, 1958, **4**, 296) is used with incorporation of a step from that of Ackerman and Myers (*Anal. Abstr.*, 1960, **7**, 4956), *viz*, removal of the protein pellicle.
H. F. W. KIRKPATRICK

5203. **Electrophoretic sub-fractionation of human serum lipoproteins by use of TEB buffer.** F. R. Sonnino and P. P. Gazzaniga (Inst. of Gen. Path., Univ. of Rome, Italy). *Clin. Chim. Acta*, 1961, **6** (2), 295–297 (in English).—A buffer consisting of tri(hydroxymethyl)methylamine (0·5M), EDTA (0·02M) and boric acid (0·07M), with a pH of 8·8 and $\mu = 0{\cdot}5$, is recommended for paper electrophoresis of serum lipoproteins. Its use permits sharper separation and greater resolution of the fractions. H. F. W. KIRKPATRICK

5204. **Electrophoresis of lipoproteins, pre-stained with Sudan black B dissolved in a mixture of dioxan and ethanediol.** A. Zakey and M. Gros (Surgical Clinic, Med. Faculty of Ljubljana, Yugoslavia). *Clin. Chim. Acta*, 1960, **5** (6), 947 (in English).—A saturated soln. of the dye (C.I. Solvent Black 3) in dioxan (1 vol.) is diluted with ethanediol (4 vol.) and centrifuged. The dye soln. (0·01 ml) is added to serum (1 ml), and after standing for 5 hr. at 37° and 12 hr. at 4° the mixture is centrifuged at 4000 r.p.m. for 20 min. Elution of the stained areas after electrophoresis is carried out with 96% ethanol - 98 to 100% acetic acid (4:1). H. F. W. KIRKPATRICK

5205. **New exchanger for the chromatographic fractionation of nucleic acids.** K. C. Smith, S. Rebhun and H. S. Kaplan (Dept. of Radiology, Stanford Univ. Sch. of Med., Palo Alto, Calif., U.S.A.). *Anal. Biochem.*, 1960, **1** (3), 202–212.—A tertiary aminoalkyl ether of starch (Cato-2), which acts as an anion exchanger, is a suitable medium for the chromatography of nucleic acids, with a capacity for deoxyribonucleic acid of 3 mg per g and for ribonucleic acid of 9 mg per g. Details of the preparation of columns are given and typical elution patterns for samples of nucleic acids from mouse- and calf-thymus glands are shown. The advantages of Cato-2 are, that it permits relatively rapid flow-rates to be employed while maintaining good sensitivity, and that the biological activity of the fractions is apparently unimpaired by passage through the columns. J. R. HUDSON

5206. **A colorimetric determination of purine and pyrimidine deoxyribonucleosides and their phosphorylated derivatives.** L. Grossman and J. Greenlees (Graduate Dept. of Biochem., Brandeis Univ., Waltham, Mass., U.S.A.). *Anal. Biochem.*, 1961, **2** (3), 189–204.—The colorimetric method of Waravdekar and Saslaw (*Anal. Abstr.*, 1957, **4**, 3067; *Ibid.*, 1960, **7**, 1848) for the determination of 2-deoxyribose in free aldehyde form may be used for differential quant. analysis of mixed purine and pyrimidine deoxyribonucleosides, since the former are quant. cleaved by 5% trichloroacetic acid at 100°, liberating free 2-deoxyribose, whereas the latter require preliminary catalytic hydrogenation to reduce the pyrimidine-ring moiety. The technique described is applicable to the determination of enzymic transglycosidations between a pyrimidine deoxyribonucleoside and a free purine, or vice versa. Thymidine and deoxycytidine may be further distinguished by a differential acid-hydrolysis procedure. Triphosphate derivatives of purine and pyrimidine deoxyribonucleosides may be determined after pretreatment with potato apyrase and prostatic phosphomonoesterase, while monophosphate derivatives require only treatment with the latter enzyme. The procedure has been used for the differential determination of these compounds. D. G. Moss

5207. **Note on the Searcy - Bergquist cholesterol reaction.** R. E. Bowman and R. C. Wolf (Primate Lab., Univ. of Wisconsin, Madison, U.S.A.). *Clin. Chim. Acta*, 1961, **6** (1), 146–148.—This reaction of cholesterol with an iron reagent (*Anal. Abstr.*, 1960, **7**, 4972) has been investigated for variability of results due to reagent quality and quantity, and interference by other substances. It is concluded that this reaction is probably no more specific than the Zak reaction (*cf.* Zlatkis *et al.*, *J. Lab. Clin. Med.*, 1953, **41**, 486), and that the validity of the method must be established by further tests, comparing the results with those of an established procedure. H. F. W. KIRKPATRICK

5208. **Method for measurement of cholesterol in blood serum.** G. V. Mann (Dept. of Surgery, Biochem. and Med., Vanderbilt Univ. Sch. of Med., Nashville, Tenn., U.S.A.). *Clin Chem.*, 1961, **7** (3), 275–284.—The method described combines the saponification and extraction procedure of Abell *et al.* (*J. Biol. Chem.*, 1952, **195**, 357) with the colour reaction with an iron reagent, developed by Zlatkis *et al.* (*J. Lab. Clin. Med.*, 1953, **41**, 486). H. F. W. KIRKPATRICK

5209. **Rapid modification of the Pearson reaction for total serum cholesterol.** G. L. van Boetzelaer and H. A. Zondag (St. Elisabeth's Hosp., Haarlem, Netherlands). *Clin. Chim. Acta*, 1960, **5** (6), 943–944 (in English).—Dissolve toluene-*p*-sulphonic acid (45 g) in acetic anhydride (600 ml) and add glacial acetic acid (400 ml). Mix 35 ml of this soln. with conc. H_2SO_4 (7 ml) and cool to room temp. This mixture should not be prepared in greater quantity for reasons of safety, and care should be taken in its disposal after use. To the mixture (5 ml) add the serum (0·1 ml), mix by blowing a current of air through the pipette for at least 5 sec., and rinse the pipette twice with the mixture. Measure the max. extinction at 615 mμ (usually 5 to 10 min. after mixing) and read the cholesterol concn. from a graph prepared by adding cholesterol to serum and treating by the method described above. H. F. W. KIRKPATRICK

5210. **Determination of free and esterified cholesterol by column chromatography. Normal blood values for umbilical cord, children and adults.** G. Maggioni, E. Bottini, C. Ioppolo and A. Signoretti (Ist. Clin. Pediat., Univ., Roma, Italy). *Diagn., Lab. e Clin.*, 1960, **16** (7), 289–295.—Free and esterified cholesterol in plasma are determined by the Liebermann - Burchard reaction after chromatographic separation. Extract the plasma (1 ml) by the method of Paget and Peirrart (*Bull. Soc. Chim. Biol.*, 1939, **21**, 528 and 537). Evaporate an aliquot to dryness and dissolve the residue in $CHCl_3$ - light petroleum (1:20). Pass the soln. through a column of alumina (5 g in a tube of 10 mm diam.). Elute first with $CHCl_3$ - light petroleum (1:9) (50 ml) collecting in 10-ml fractions. Continue elution with $CHCl_3$ - ethyl acetate (1:1) (70 ml), collecting similarly. Evaporate each fraction to dryness, dissolve the residue in $CHCl_3$ (7 ml) and carry out the Liebermann - Burchard reaction (Schoenheimer and Sperry, *J. Biol. Chem.*, 1934, **106**, 745). The esterified cholesterol is found in fractions 2 to 4 and the free cholesterol in fractions 7 to 10. Separation is complete. Coefficients of variation were—esterified cholesterol ±6·95%, free cholesterol ±5·46%, ratio of esterified to free ±8·59%. J. I. M. JONES

5211. **Determination of oestrogens. I. Determination of total oestrogens in plasma.** G. W. Oertel (Dept. of Biol. Chem., Coll. of Med., Univ. of Utah, Salt Lake City, U.S.A.). *Clin. Chim. Acta*, 1961, **6** (2), 237–241 (in German).—Plasma is extracted with ethanol and the extract, after acid hydrolysis, is extracted with ethyl acetate. The phenolic steroids are separated, and purified by chromatography on alumina. The Ittrich modification of the Kober reaction (*Anal. Abstr.*, 1959, **6**, 3136) is used for colorimetric determination. The method is sensitive only to concn. of oestrogen of >0·1 μg per 10 ml of plasma, and in non-pregnancy plasma at least 20 ml of sample is required.

II. Determination of oestrone, oestradiol and oestriol in pregnancy plasma. G. W. Oertel. *Ibid.*, 1961, **6** (2), 242–245.—The total oestrogens obtained in Part I are separated by paper chromatography on acetylated paper and then purified by chromatography on alumina. The oestrogens are determined as described above.

H. F. W. KIRKPATRICK

5212. **Chemical determination of oestrogenic hormones in blood, milk and colostrum.** G. Ittrich (Hormonlab., Univ.-Frauenklinik der Charite, Berlin). *Hoppe-Seyl. Z.*, 1960, **320**, 103–110.—A method is described for determination of oestrone, oestradiol and oestriol by the Kober reaction with *p*-nitrophenol. Colorimetric and fluorimetric procedures are given. The method is specific and reproducible, and needs only 0·04 μg of oestrogen in the sample. Recoveries of 72·5 ±5% for oestrone, 73·6 ±6% for 17β-oestradiol, and 74·9 ±4·5% for oestriol were obtained. Determinations have been carried out on venous blood, cord blood and colostrum. F. POWELL

5213. **Studies on the analysis of steroid hormones by high-voltage paper electrophoresis. IV. The electrophoretic patterns of the crude conjugated oestrogens in urine.** Toshio Iso (School of Med., Tokyo Medical and Dental Univ., Tokyo). *J. Japan. Biochem. Soc.*, 1959, **31** (1), 27–33.—A crude conjugated oestrogen fraction was prepared by butanol extraction of the urine of pregnant females, and separation by paper electrophoresis was effected at 40 kV in formic acid - acetic acid solution. The electrophoretic pattern was detected by fluorescence under u.v. light followed by a bioassay of the oestrogenic activity. Eight conjugated oestrogens were detected by this method.

S. NATORI

5214. **Note on the chemical determination of 16-epioestriol.** W. Nocke (Chem. Abt., Chirurg. Univ.-klinik, Bonn-Venusberg, Germany). *Clin. Chim. Acta*, 1961, **6** (3), 449–452 (in English).—In the method of Brown (*Anal. Abstr.*, 1955, **2**, 3161), the aq. fraction which contains the oestriol and 16-epioestriol [oestra-1,3,5(10)-triene-3,16β,17β-triol] (**I**) is methylated by a new procedure and chromatographed on alumina. Alternatively, **I** is converted into its acetonide and is separated by partition between $CHCl_3$ and dil. alkali, chromatographed as the acetonide and determined by a modified Kober reaction.

H. F. W. KIRKPATRICK

5215. **Determination of dehydroepiandrosterone and total neutral 17-oxosteroids in human plasma.** B. Hudson and G. W. Oertel (Coll. of Med., Univ., Salt Lake City, Utah, U.S.A.). *Anal. Biochem.*, 1961, **2** (3), 248–262.—After preliminary purification as described by Kornel (*Anal. Abstr.*, 1960, **7**, 3927), the plasma extract, in 5% H_2SO_4, is extracted with ethyl acetate and this soln. is solvolysed overnight at 38° to 40°, while the remaining H_2SO_4 solution is hydrolysed at 100° and extracted with a further portion of ethyl acetate. The extracts are combined, washed with 5N-NaOH and water, and evaporated to dryness. Separate determinations of dehydroepiandrosterone (3β-hydroxyandrost-5-en-17-one) (by the ethanol - H_2SO_4 reaction) and total 17-oxosteroids (by the Zimmermann reaction) are carried out. Normal plasma levels by this method are significantly

higher than those previously reported, and this is believed to be due to the improved hydrolysis technique. D. G. Moss

5216. **Determination of steroids by paper-strip elution chromatography.** D. M. Abelson, P. K. Bondy and J. Piskorski (Yale Univ. School of Med., New Haven, Conn., U.S.A.). *J. Chromatography*, 1961, **5** (4), 332–335 (in English).—The chromatographic chamber consists of a glass cylinder (6 in. in diam. × 12 long) with a 1-in. hole in the base. This is closed with a polyethylene stopper, through which passes a small funnel. The paper is a 25-cm strip of washed Whatman 3MM or No. 1 paper, pointed at the lower end. The distance from origin to tip is 19 cm. Solvent drips off the pointed tip through the funnel into test-tubes (10 mm × 75 mm), mounted on a time-operated fraction collector. For steroids, 5 to 10 μg of each is used and separated with the toluene - 75% methanol system. Twenty-four fractions of 0·5 ml are collected, the solvent is removed *in vacuo* and the steroids are determined by alkaline fluorimetry in K t-butoxide (Abelson *et al.*, *Anal. Abstr.*, 1956, **3**, 209). Separations of corticosterone, cortisone and cortisol are illustrated. In 4 experiments, the recovery of cortisol was 92 ±11%. G. Russell

5217. **Paper adsorption chromatography of the acetates of weakly polar steroids.** J. R. Pasqualini and M. F. Jayle (Lab. Biol. Chem., Fac. Med., Paris). *J. Chromatography*, 1961, **5** (4), 336–340 (in French).—Monoacetates of hydroxysteroids and di-acetates of dihydroxysteroids are separated on Whatman No. 1 paper with petroleum spirit, heptane, 2,2,4-trimethylpentane, methylcyclohexane or decalin as solvent; 3 to 8 μg of each acetate (prep. described) in 4 to 10 μl of ethyl acetate - acetone (1:1) is used. The paper is equilibrated with solvent for 24 hr. and precautions to avoid part of the sample remaining at the origin are mentioned. The first 4 solvents are allowed to run for 3 to 4 hr. (16 to 27 cm) and the decalin for 8 hr. (28 cm). α-Ketolic steroids are detected with blue tetrazolium and 17-oxosteroids with the Zimmermann reagent. R_F values relative to selected standards are listed. G. Russell

5218. **Critical study of the method of Reddy, and modifications thereof, for the determination of urinary 17α,21-dihydroxy-20-oxosteroids.** M. F. Hebbelynck (Lab. de Biochim., Fondation Méd. Reine Elisabeth, Brussels, Belgium). *Clin. Chim. Acta*, 1960, **5** (6), 862–878 (in French).—Recommended modifications are a specially purified n-butanol prepared by distillation from 2,4-dinitrophenylhydrazine and H_2SO_4; saturation of this solvent with H_2O and Na_2SO_4 before its use for the extraction of urine and preparation of blanks and standards; carrying out the Porter - Silber colour reaction at 25° for 20 hr. with 20N-H_2SO_4; and reading the extinction at 406 mμ.

H. F. W. Kirkpatrick

5219. **Paper-chromatographic determination of steroids in adrenal cortical extracts.** G. Cavina, E. Cingolani and L. Tentori (Lab. Biol., Ist. Sup. Sanità, Roma, Italy). *R.C. Ist. Sup. Sanit.*, 1960, **23** (12), 993–1007.—Methods for the determination of steroids in adrenal cortical extracts are reviewed and from them a general method is developed based on the chromatographic systems of Burton *et al.* (*J. Biol. Chem.*, 1951, **193**, 769), Bush (*Biochem. J.*, 1952, **50**, 370) and Eberlein and Bongiovanni (*Arch. Biochem. Biophys.*, 1955, **59**, 90). Location of the individual steroids is effected with u.v. light, and eluates from the chromatograms are assayed with blue tetrazolium reagent, or, in the case of aldosterone, by the absorption at 288 mμ in conc. H_2SO_4, or at 280 mμ in 100% H_3PO_4. Cortisol, cortisone, their tetrahydro-derivatives, corticosterone, aldosterone, 11-dehydrocorticosterone and 17-hydroxy-11-dehydrocorticosterone can be determined, and other characteristic steroids present in small amounts in the amorphous fraction of the extracts can be recognised. Experiments with known mixtures show that recovery from the chromatogram is 87 to 95%, the different compounds being assessed as cortisol. It is suggested that the method may lead to a useful correlation of the composition of extracts with the biological maintenance assay of Bomskow and Bahnsen (*Arch. exp. Path. Pharmak.*, 1935, **178**, 1). J. I. M. Jones

5220. **Photometric micro-determination of serum lipase with a phenyl laurate substrate.** A. Saifer and G. Perle (Isaac Albert Res. Inst., Jewish Chron. Dis. Hosp., Brooklyn, N.Y., U.S.A.). *Clin. Chem.*, 1961, **7** (2), 178–184.—The method of Raderecht and Moskau (*Anal. Abstr.*, 1959, **6**, 4119) has been modified for application to blood serum.

H. F. W. Kirkpatrick

5221. **Measurement of serum lactic dehydrogenase.** H. A. Ells (Oklahoma Med. Res. Inst., Oklahoma City, U.S.A.). *Clin. Chem.*, 1961, **7** (3), 265–274.—A colorimetric method is described in which phenazine methosulphate is used as intermediate in electron transfer between reduced diphosphopyridine nucleotide and 2,6-dichlorophenolindophenol (*cf. Anal. Abstr.*, 1960, **7**, 3935).

H. F. W. Kirkpatrick

5222. ***p*-Amino-*NN*-dimethylaniline as substrate for the colorimetric determination of peroxidase in plants.** T. Cervigni and M. Giacomelli (C.N.E.N. Lab. di Genetica Vegetale, Centr. Stud. Nucl. d. Casaccia, Roma, Italy). *Ital. J. Biochem.*, 1961, **10** (2), 65–73.—Peroxidase in plant material is determined by measuring the rate of oxidation of *p*-amino-*NN*-dimethylaniline hydrochloride (**I**) by the enzyme in the presence of H_2O_2. This is effected by measuring the extinction at 520 mμ of the reaction mixture, which develops a red colour, deepening with time. *Procedure*—Grind the plant material (1 g) with acetone (10 ml) pre-cooled to −15°. Filter off and wash the undissolved powder with acetone (100 ml) and dry in a desiccator. Make 50 mg of the powder into a paste with H_2O, filter and use the filtrate (1 ml) with 4 ml of Sørensen buffer (pH 6·3). To this mixture add $1·7 \times 10^{-4}$M-**I** (1 ml) and incubate for 10 min. at 20°. Add $2·5 \times 10^{-4}$M-H_2O_2 (0·1 ml), mix and read the extinction at 520 mμ after 10 and 60 to 80 sec. Calculate the change in extinction per sec. and from the mol. extinction coeff. (6596 at 520 mμ) calculate the reaction constant in μmoles of **I** oxidised per sec. as a measure of peroxidase activity. Reproducibility is good. Acetone-extracted powder is preferable to homogenates of the material, which may contain inhibitors only partially removed by dialysis.

J. I. M. Jones

5223. **Determination and characterisation of aminotripeptidase in normal human serum.** R. J. Haschen (II Med. klinik d. Martin-Luther-Univ., Halle/Saale, Germany). *Clin. Chim. Acta*, 1961, **6** (3), 316–321 (in German).—The biuret method of London *et al.* (*Anal. Abstr.*, 1957, **4**, 2349) is modified, as is Haschen's previous method (*Anal. Abstr.*, 1957, **4**, 2348), by using quant. paper chromatography. These methods are suitable for determining the enzyme, which has optimum activity at pH 6·8 and requires certain bivalent metal ions (*e.g.*, Co^{2+}) for activation.
H. F. W. KIRKPATRICK

5224. **Determination and characterisation of leucineaminopeptidase in normal human serum.** R. J. Haschen (II Med. Klinik d. Martin-Luther-Univ., Halle/Saale, Germany). *Clin. Chim. Acta*, 1961, **6** (3), 322–325 (in German).—A microdiffusion method and a quant. paper-chromatographic method are described in which leucinamide is used as substrate. The enzyme has optimum activity at pH 8·0 in the presence of Co^{2+}; Mn^{2+} and Mg^{2+} inhibit the activity.
H. F. W. KIRKPATRICK

5225. **An ultrasensitive assay of β-galactosidase with carbon-14-labelled o-nitrophenyl-β-D-galactoside.** H. Noll and J. Orlando (Dept. of Microbiol., School of Med., Univ., Pittsburgh, Pa., U.S.A.). *Anal. Biochem.*, 1961, **2** (3), 205–215.—The colorimetric determination of β-galactosidase, with *o*-nitrophenyl-β-D-galactoside (**I**) as substrate, was modified to give a 5-fold increase in sensitivity by extracting the liberated *o*-nitrophenol from the acid reaction mixture with 2,2,4-trimethylpentane, and re-extracting into a smaller vol. of aqueous alkali. The extinction was measured at 415 mμ, with an ultra-micro cell. This technique also eliminates turbidity and yellow background colour derived from biol. fluids. A further considerable increase in sensitivity was obtained by using radioactive **I** labelled with ^{14}C in the benzene ring, and measuring the radioactivity extracted into 2,2,4-trimethylpentane. This latter technique enabled hydrolysis rates of less than 10^{-10} mole of **I** per 20 min. to be determined with an accuracy of ±10%. The synthesis of labelled **I** is described.
D. G. Moss

5226. **Serum β-glucuronidase assay by the phenolphthalein mono-β-glucuronide method.** G. Goldstein (Mt. Sinai Hosp., New York, N.Y., U.S.A.). *Clin. Chem.*, 1961, **7** (2), 136–142.—Comparative assays showed that low results for enzyme activity resulted from protein pptn. with trichloroacetic acid and heat coagulation. No loss occurs if acetone is used for pptn. Optimum activity of the enzyme is obtained at pH 4·1 to 4·2 in acetate buffer at 37°. The assay is most conveniently performed without protein pptn.
H. F. W. KIRKPATRICK

5227. **The determination of tartrate-inhibited phosphatase in serum.** K. Jacobsson (Centrallasarettet, Växjö, Sweden). *Scand. J. Clin. Lab. Invest.*, 1960, **12** (4), 367–380.—A method is described for determining tartrate-inhibited phosphatase in serum with the use of the chromogenic substrate, *p*-nitrophenyl disodium phosphate.
P. PEDERSEN

5228. **Spectrofluorimetric determination of acid phosphatase activity.** D. M. Campbell and D. W. Moss (Postgrad. Med. Sch., Ducane Rd., London, W.12). *Clin. Chim. Acta*, 1961, **6** (3), 307–315.—In the method described, 1-naphthyl phosphate is used as substrate (*cf. Anal. Abstr.*, 1960, **7**, 2418), with an incubation time of 15 min. at 37°. This substrate is shown to be not entirely specific for the prostatic enzyme. H. F. W. KIRKPATRICK

5229. **Determination of phosphofructokinase in erythrocytes.** L. Mirčevová and J. Vosyková (Inst. for Haematology and Blood Transfusion, Prague). *Coll. Czech. Chem. Commun.*, 1961, **26** (5), 1469–1472 (in German).—The phosphofructokinase activity of erythrocytes haemolysed with water is determined photometrically by a method similar to that for aldolase activity (Sibley and Lehninger, *J. Biol. Chem.*, 1949, **177**, 859) as modified by Bruns (*Biochem. Z.*, 1954, **325**, 156). The soln. is incubated at pH 7·3 for 15 min. with soln. of fructose 6-phosphate, adenosine triphosphate, $MgCl_2$ and hydrazine sulphate. The soln. is de-proteinised with trichloroacetic acid, and the triose phosphate formed is determined by measurement of the extinction at 530 mμ after addition of 2,4-dinitrophenylhydrazine and NaOH soln. The limits of error are ±11%. J. P. STERN

5230. **Biochemical method for the determination of oxytocinase and its clinical value.** R. Klimek and M. Pietrzycka (I Klinik f. Frauenkrankheiten u. Geburtshilfe, Univ. Krakow, Poland). *Clin. Chim. Acta*, 1961, **6** (3), 326–330 (in German).—The method of Tuppy and Nesvadra (*Monatsh. Chem.*, 1957, **88**, 977; *Arch. Gynäkol.*, 1959, **191**, 442) is investigated and the chemical and clinical results are confirmed. In pregnancy, enzyme activity increases during the third month and rises continuously until delivery. Activity is also detectable in the urine of non-pregnant women and men. Details of the method are given.
H. F. W. KIRKPATRICK

See also Abstracts—**4918**, Papers from I.U.P.A.C. Congress, Lisbon, 1956. **4922**, Determination of choline. **5023**, Cobalt in biological material. **5058**, Determination of adenosine triphosphate and DPN+. **5059**, Assay of glucosidases. **5240**, Phenothiazine drugs in urine. **5300**, Use of gel filtration. **5302**, Standard for pH of blood. **5308**, Apparatus for elution of steroids. **5331**, Flame photometry of K.

Pharmaceutical analysis

5231. **Ultra-violet spectrophotometry and its application to the evaluation of pharmaceutically active compounds.** J. Kráčmar (Staatl. Inst. f. Arzneimittelkontr., Prague, Czechoslovakia). *Pharmazie*, 1961, **16** (7), 341–350.—A review with 270 references to papers published up to October 30th, 1960. N. E.

5232. **Analytical study of alkaloids by the hydroxylamine - ferric chloride colour reaction.** D. Vincent and H. Schwal (Lab. de Mat. Méd., Fac. de Méd. et de Pharm., Toulouse, France). *Ann. Pharm. Franç.*, 1961, **19** (2), 73–80.—The method of Hestrin (*J. Biol. Chem.*, 1949, **180**, 249), in which a red colour with hydroxylamine - $FeCl_3$ is developed by certain esters through the formation of a hydroxamic acid - $FeCl_3$ complex, is applied to a

number of alkaloids. *Procedure*—The alkaloid (5 mg) in aq. HCl soln. (1 ml) is treated with 0·4 ml of a mixture of 13·9% hydroxyammonium chloride (1 part) and 3·5M-NaOH (2 parts). After 1 min., 0·3 ml of 4M-HCl and 0·2 ml of 0·37M-$FeCl_3$ in 0·1M-HCl, and 5 ml of water are added. The extinction is determined at 520 mμ. Among ester alkaloids a particularly intense reaction (which is contributed to by the colour given with NaOH and $FeCl_3$ without hydroxylamine) is given by physostigmine and geneserine. A moderate coloration is given by diamorphine, cocaine and a number of other ester alkaloids, and a much lower coloration by veratrine, pseudo-aconitine, genohyoscine, genatropine, genohyoscyamine, hyoscine, atropine, hyoscyamine, homatropine and delphinine. No coloration is given by yohimbine, corynanthine, serpentine and raubasine. Among alkaloids which are not esters, the phenol function in some alkaloids gives rise to some colour, but pilocarpine gives a good colour reaction, due probably to the opening of the lactone ring. Applications are discussed.

E. J. H. Birch

5233. **Paper-chromatographic separation of mixtures of alkaloids on succinylcellulose papers.** F. Micheel and W. Leifels (Org.-chem. Inst., Univ. Münster, Germany). *Mikrochim. Acta*, 1961, (3), 444–448 (in German).—Succinylcellulose paper, prepared by a method described previously (*Chem. Ber.*, 1958, **91**, 1212), was used for the separation of the principal alkaloids found in extracts of opium, nux vomica, belladonna and hyoscyamus. The solvent was a mixture of ethyl acetate, formic acid and water (10:1:3). The opium chromatograms were developed in a second dimension with dioxan - formic acid - water (180:1:19). Several other groups of alkaloids were examined.

R. M. Rowley

5234. **Spectrophotometric differentiation and determination of morphine derivatives.** E. Vidic (Inst. f. gerichtl. u. sozial. Med., Freie Univ., Berlin). *Arzneimittel-Forsch.*, 1961, **11** (4), 408–413.—Heating with 72% H_2SO_4 at 85° during 1 hr. transforms morphine, codeine and dihydrocodeine, all containing an alcoholic hydroxyl group, into products which give u.v. spectra with distinctive individual characteristics. The extinction coefficients of the new maxima are 3·5 to 9 times those of the original substances in acid solution. Ketonic alkaloids of the series, such as morphinones and codeinones, are much less affected and do not show a notable change in the spectra. The treatment with H_2SO_4 makes the spectrophotometric identification of morphine derivatives more sensitive, and, in conjunction with paper chromatography, also permits a quantitative determination.

M. Sulzbacher

5235. **Behaviour of alkaloidal salts on columns of aluminium oxide. IX. Assay and checking of purity of quinine and its salts.** H. Böhme, R. Neidlein and E. Tauber (Pharm.-chem. Inst., Univ. Marburg/Lahn, Germany). *Arch. Pharm., Berlin*, 1961, **294** (5), 315–321.—Quinine (**I**) salts are decomposed on alumina and the liberated **I** is determined by titration with 0·1N-HCl, by isolation and weighing, or by bromimetric titration when hydroquinine (**II**) does not interfere. *Procedure*—Dissolve **I** hydrochloride (≃0·1 g) in 90% aq. acetone (30 ml) and percolate the soln. through a column of 4 g of activated alumina slurried with aq. acetone. Elute with aq. acetone (3 × 10 ml), dilute the eluate with water (100 ml) and titrate with 0·1N-HCl, with methyl red - methylene blue as indicator. **I** sulphate is determined similarly, with 80% aq. acetone as solvent. To determine **I** base, dissolve a sample (0·4 g) in methanol (50 ml), dilute an aliquot (10 ml) with acetone (25 ml) and water (80 ml), then titrate as before. To determine the content of **II** hydrochloride in **I** hydrochloride, dissolve the isolated base or a fresh sample (≃0·1 g) in N-HCl and methanol (10 ml), and treat in the dark for 10 min. at 20° with 10 ml of 0·1N-Br. Add methanol (20 ml) and 15% KI soln. (3 ml), then titrate with 0·1N-$Na_2S_2O_3$, with starch as indicator. Perform a blank titration and calculate **II** hydrochloride by difference.

A. G. Cooper

5236. **The characterisation of quinotoxine in the presence of quinine in salts of quinine.** J. Soleil and [Mme] G. Bellot (Lab. de Chim. Org. et de Pharm. Chim., Fac. de Pharm., Nancy, France). *Ann. Pharm. Franç.*, 1961, **19** (1), 37–43.—Methods for detecting quinotoxine in the presence of quinine are examined and discussed; the only ones which are reliable in the presence of much larger quantities of quinine are the use of Fröhde's sulphomolybdic acid reagent and Lafon's sulphoselenous acid reagent. *Procedure*—The quinine salt (0·6 g) is dissolved in 10 ml of water with sufficient H_2SO_4 to complete the dissolution. To the soln. is added 10% NaOH soln. until it is alkaline to litmus; the aq. layer is extracted with 3 × 20-ml portions of $CHCl_3$ and, after evaporation and making up to 8 ml with $CHCl_3$, the soln. is treated with 2 ml of either of the reagents. With Fröhde's reagent the presence of quinotoxine is indicated by the development after 12 hr. of a reddish-brown ring at the interface of the two liquids (in the absence of quinotoxine a greenish-yellow ring is formed.) With Lafon's reagent the H_2SO_4 part of the soln. is coloured pink to red in the presence of quinotoxine after 15 to 20 min. (yellow in its absence), or after 12 hr. the ring at the interface of the liquids is red - brown in the presence of quinotoxine (yellow in its absence). One part of quinotoxine in 1000 parts of quinine can be detected.

E. J. H. Birch

5237. **Colorimetric determination of reserpine.** Naochika Nakamura and Toshiko Yoshida (Takeda Pharm. Ind., Higashiyodogawa-ku, Osaka, Japan). *Ann. Rep. Takeda Res. Lab.*, 1959, **18**, 25–29.—An acetic acid soln. (1 ml) of reserpine (50 to 500 μg) was heated with xanthhydrol soln. in HCl in a water bath for 10 min. Unchanged xanthhydrol was extracted from the cooled soln. with benzene, the aq. layer was bubbled with air, and the extinction was measured at 500 mμ to determine the content of reserpine.

S. Natori

5238. **A micro-fluorimetric method for the determination of agmatine.** V. H. Cohn, jun., and P. A. Shore (National Heart Inst., Bethesda, Maryland, U.S.A.). *Anal. Biochem.*, 1961, **2** (3), 237–241.—After protein pptn. by addition of $HClO_4$ to a final concn. of 0·4N, and centrifuging, an aliquot of the supernatant liquid is made alkaline by adding 0·5 ml of 5N-NaOH, and is extracted with 10 ml of butanol. The organic phase, which contains the agmatine, is separated and washed with 0·1N-NaOH satd. with NaCl to remove residual amounts of arginine, which would otherwise be estimated with the agmatine. An 8-ml portion of the butanol soln. is now mixed with 15 ml of

heptane, and extracted with 3 ml of 0·1N-HCl. After centrifugation, 2 ml of the HCl soln. is mixed with 0·4 ml of N-NaOH and 0·1 ml of freshly prepared phthalaldehyde soln. (10 mg in 100 ml of methanol). After 10 min. the resulting fluorescence is measured at 470 mμ (activation wavelength 355 mμ). The fluorescence intensity is proportional to the agmatine concn. over the range 0·05 to 5 μg per ml. D. G. Moss

5239. **Determination of moisture-retaining constituents in tobacco products.** H. Puschmann and J. E. Miller (Farbwerke Hoechst A.-G., Gendorf/Oberbayern, Germany). *Z. LebensmittUntersuch.*, 1961, **114** (4), 297–301.—The chief moisture-retaining constituents are diethylene glycol, 1,2-propylene glycol, 1,3-butylene glycol and glycerol. These were converted into the corresponding esters with acetic anhydride at 120°. Details are given of two methods, (*i*) extraction of the sample (10 g) with $CHCl_3$ before esterification and (*ii*) direct esterification of the sample (2·5 g) in a closed system. For tobaccos containing 0·5 to 10% of moisture-retaining constituents, method (*ii*) is the better. The esters were extracted and determined by gas chromatography. Tetraethylene glycol dimethyl ether and triethylene glycol dimethyl ether were used as internal standards. S.C.I. Abstr.

5240. **Method for the chromatographic detection of penicillin and related compounds and of penicillinase.** P. H. A. Sneath and J. F. Collins (Nat. Inst. for Med. Res., Mill Hill, London, England). *Biochem. J.*, 1961, **79** (3), 512–514.—The test is based on the reaction of iodine with penicilloic acids. The intact penicillin mol. is not oxidised by iodine, but, after the β-lactam ring is opened, the resulting penicilloic acid reacts rapidly with 8 to 9 equiv. of iodine. The paper is sprayed with a starch - iodine soln.; compounds with an open β-lactam ring immediately give pale spots on a dark-blue background, while compounds with a closed β-lactam ring do not react. The paper is then sprayed with a soln. of penicillinase - starch - iodine, when additional pale spots appear at the site of penicillins whose β-lactam ring is opened by the enzyme. The method can also be used for the detection of penicillinase by use of a soln. of penicillin - starch - iodine. J. N. Ashley

5241. **Automatic determination of penicillin in fermentation broth. An improved iodimetric assay.** R. R. Goodall and R. Davies (I.C.I. Ltd., Pharm. Div., Alderley Park, Ches., England). *Analyst*, 1961, **86**, 326–335.—The automatic method of Ferrari *et al.* (*Anal. Abstr.*, 1960, **7**, 2431) is modified. By means of a pair of self-levelling dilution pipettes, arranged in series, the reactants (penicilloic acid and iodine) are brought within the range of the recording colorimeter employed, in which the excess of iodine is measured as its blue complex with starch. Penicillinase is conveniently included in the diluent so that the automatic stage is confined to iodination, mixing with starch soln. and recording the extinction. Standard soln. of penicilloic acid are prepared by alkaline hydrolysis of phenoxymethylpenicillin, but in the sample soln. the hydrolysis is effected with penicillinase. The coeff. of variation of the assay is 2 to 2·5%.
A. O. Jones

5242. **The micro-determination of chloramphenicol and its use in the preservation of crab meat.** Akira Ito and Koichi Okamoto (Takamine Lab., Sankyo Co., Ltd., Shinagawa-ku, Tokyo). *Ann. Rep. Takamine Lab.*, 1959, **11**, 104–112.—An aq. soln. (85 ml) containing chloramphenicol was divided into two parts, one being used for the determination and the other as a control. After the addition of 0·25N-NaOH (20 ml), the soln. was stirred with 0·15% $Na_2S_2O_4$ soln. (6·25 ml) for 15 min. The amino-compound thus formed was diazotised with 0·25% $NaNO_2$ soln. (6·25 ml) and 2N-HCl (12·5 ml). After the addition of 0·5% sulphamic acid soln. (62·5 ml), the soln. was treated with Tsuda's reagent [*N*-(2-diethylaminoethyl)-1-naphthylamine oxalate] and set aside at room temp. for 2 hr. The soln. was treated with KCl (30 g) and extracted with butanol (100 ml) for 5 min. The extinction of the butanol layer was determined at 574 mμ. S. Natori

5243. **Quantitative determination of kanamycin and its separation from other antibiotics.** F. Cotta-Ramusino, R. Intonti and A. Stacchini (Ist. Sup. di Sanità, Roma). *Ann. Chim., Roma*, 1961, **51** (3–4), 290–299; *R.C. Ist. Sup. Sanit.*, 1960, **23** (12), 1048–1057.—Kanamycin in soln. is determined by heating with a soln. of ninhydrin (100° for 25 min.) and measuring the extinction at 575 mμ. In the presence of other antibiotics, *e.g.*, neomycin, also reacting with ninhydrin, the soln. is applied to a column of cation-exchange resin, the kanamycin is eluted under standardised conditions, the ninhydrin reaction is carried out on fractions of the eluate and the concn. of kanamycin determined from the extinction - elution curve.
L. A. O'Neill

5244. **Paper chromatography of barbiturates using formamide and ethanediol as stationary phases.** F. Dybing (Dept. of Pharmacol., Univ. Oslo, Norway). *Scand. J. Clin. Lab. Invest.*, 1960, **12** (3), 333–335.—Chloroform satd. with stationary phase is used as the mobile phase. R_F values for 18 barbituric acid derivatives are given. The spots are located in u.v. light after being sprayed with NaOH soln. Unsaturated and saturated compounds may be distinguished by spraying with 0·02N-$KMnO_4$. P. Pedersen

5245. **Identification of sulphadimidine and some other sulphonamides with Bouchardat's reagent.** H. M. Romijn. *Pharm. Weekbl.*, 1961, **96** (7), 245–248.—Sulphadimidine (**I**) can be distinguished from 19 other sulphonamides by the use of Bouchardat's reagent (iodine in KI soln.), which gives a black ppt. with **I**. In mixtures with these sulphonamides, 10% of **I** can be identified in this way, but in mixtures with sulphathiazole a concn. of at least 33% is required. **I** and seven other sulphonamides also give characteristic microchemical reactions with Bouchardat's reagent. (8 illustrations.) M. J. Maurice

5246. **Selective extraction and colorimetric determination of 10-dialkylaminoalkylphenothiazines.** A. Z. Kotionis (Athens Dye Works, Moschaton, Greece). *Arzneimittel-Forsch.*, 1961, **11** (2), 108–110 (in English).—These compounds (**I**) form $CHCl_3$-sol. complexes with Cl^- and with bromothymol blue (**II**). Complexes (1:1) with **II** are extracted into $CHCl_3$, decomposed with alkali and the liberated **II** is measured photometrically. Interference by other bases is prevented by extraction of the complexes with Cl^- into $CHCl_3$ before reaction with **II**. To determine **I** alone, shake the soln.

(0·1 to 1 ml, containing 50 to 300 μg of **I** hydrochloride) with $CHCl_3$ (10 ml) for 1 min., add 0·4 ml of **II** soln. [**II** (0·312 g), ethanol (4 ml) and 0·2N-NaOH (3 ml) diluted to 100 ml] and phosphate buffer of pH 7·6 (10 ml), and shake the mixture for 1 min. Separate, wash the aq. layer with $CHCl_3$ (2 ml), extract the combined $CHCl_3$ soln. into 0·01N-NaOH (10 ml), dilute the extract to 100 ml with 0·01N-NaOH, read the extinction at 615 mμ and determine **I** by reference to a standard curve. To determine **I** in the presence of interfering bases (*e.g.*, pyrilamine maleate), add 0·1N-HCl (10 ml) to a sample (0·1 to 1 ml, containing 50 to 250 μg of **I**), extract into $CHCl_3$ (2 × 5 ml) and treat the combined extracts with **II** as described above. To determine **I** in urine, mix the sample (40 ml) with N-HCl (5 ml), extract into $CHCl_3$ (2 × 40 ml), wash the combined extracts with 0·2N-NaOH (10 ml) and evaporate to 7 ml. Transfer the residue with $CHCl_3$ (3 ml) to a separating-funnel, and treat with **II** as described above. A. G. COOPER

See also Abstracts—**4918**, Papers from I.U.P.A.C. Congress, Lisbon, 1956. **4922**, Determination of penta- and deca-methonium. **5023**, Cobalt in cobalamine. **5087**, Determination of piperazine. **5136**, Identification of barbiturates. **5137**, Phenothiazine deriv. in body fluids. **5138**, *N*-(Guanidinoformimidoyl)morpholine in urine. **5139**, Phenylalanine mustard and related compounds in blood.

Food

Foods and food additives, beverages, edible oils and fats, vitamins.

5247. **Polarographic determination of copper, lead, tin and zinc in foodstuffs.** W. F. Condliffe and A. J. H. Skrimshire (H. J. Heinz Co., Ltd., Wardour Road, Harlesden, London, N.W.10). *J. Polarographic Soc.*, 1961, **7** (1), 10–14.—*Procedure*—Oxidise the sample (5 g) with HNO_3, H_2SO_4 and $HClO_4$. Dilute the acid digest to 25 ml. Determine the metals on a cathode-ray polarograph as follows: Cu^{2+} in tartaric acid - HNO_3 soln. (peak potential −0·33 V), Pb^{2+} in dipotassium hydrogen citrate - H_2SO_4 soln. (−0·69 V), Zn^{2+} in faintly alkaline $(NH_4)_2SO_4$ soln. (−1·3 V) and Sn^{2+} in ammonium bromide - HCl soln. (−0·33 V). Recoveries of the metals added to tomato paste were 97 to 107%. The method is accurate to ±7% at the 0·1 to 2·5-p.p.m. level and to ±3% at higher levels. Precautions are described to minimise the effect of vibration on the electrode assembly.
G. RUSSELL

5248. **Determination of the diastatic power of honey.** H. Hadorn (Lab. VSK, Basle, Switzerland). *Mitt. Lebensmitt. Hyg., Bern*, 1961, **52** (2), 67–103.—The existing methods are briefly reviewed and two are investigated in detail. The colorimetric method of Gothe as modified by Kiermeier and Köberlein (*Z. LebensmittUntersuch.*, 1954, **98**, 329) is shown to have several defects, particularly as a result of variations in the starch solution used, and the estimated diastatic power is 10 to 40% lower than that obtained by the method of Schade *et al.* (*Food Res.*, 1958, **23**, 446), which is modified by standardising the reagents and conditions; on the basis of 54 determinations on 18 samples of honey a coeff. of variation of 1·4% is claimed. A method of preparing soluble starch for these procedures has been developed, together with one for its control based on the "blue value" of Hadorn and Doevelaar (*Mitt. Lebensmitt. Hyg., Bern*, 1960, **51**, 1). The diastatic power and the hydroxymethylfurfuraldehyde content (Winkler's method) of 21 honey samples are tabulated; low diastatic power coupled with high hydroxymethylfurfuraldehyde content are noted for old (20 years) or overheated samples. R. CLARKE

5249. **Volumetric micro-determination of potassium with tetraphenylborate. II. Determination of potassium in milk.** F. Bermejo Martínez and A. Margalet Barral (Univ., Santiago de Compostela, Spain). *Inf. Quim. Anal.*, 1961, **15** (2), 36–40.—The sample of milk is treated with aq. Na_2WO_4 and H_2SO_4 to remove proteins, which are separated by centrifuging. The supernatant liquid is treated at pH 9 with a small amount of formaldehyde soln. and a known amount of standard Na tetraphenylborate (**I**) soln. The ppt. is filtered off and the excess of **I** is measured by titration with a standard soln. of a quaternary ammonium salt (benzylcetyldimethylammonium chloride), with bromophenol blue as indicator. L. A. O'NEILL

5250. **Determination of radio-strontium [strontium-90] in liquid milk.** G. S. Spicer (U.K.A.E.A. Research Group, C.37, Royal Arsenal, Woolwich, London, S.E.18). A.E.R.E. Analytical Method AERE-AM80, 1961. 11 pp.—After the addition of strontium and barium carriers to the milk (2 litres), HCl is added to ensure that Ca and Sr are ionic, and to precipitate the protein. The ppt. is filtered off, and calcium and strontium oxalates are pptd. from the filtrate. The ppt. is dissolved in HNO_3, and $Sr(NO_3)_2$ and $Ba(NO_3)_2$ are then pptd. by adding more acid. The pptn. is repeated and Ba is removed as $BaCrO_4$. After scavenging with $Fe(OH)_3$, yttrium carrier is added and the soln. is set aside for ^{90}Y to develop. Finally yttrium oxalate is pptd. and counted. Full details of the procedure and calculation are given and details for determining Ca and Sr in milk are appended. The initial treatment of sour milk differs in that EDTA (disodium salt) and aq. NH_3 are added so that a homogeneous soln. may be produced.
G. J. HUNTER

5251. **Volumetric determination of chloride in milk and cheese.** F. Kiermeier and C. Kayser (Forschungsanst. f. Milchwirtschaft, Weihenstephan, Germany). *Z. LebensmittUntersuch.*, 1961, **114** (3), 208–213.—Analytical data for the determination of Cl^- in milk and cheese by the methods of Hostettler *et al.* (*Mitt. Lebensmitt. Hyg., Bern*, 1948, **39**, 93) and of Drost (*Z. LebensmittUntersuch.*, 1923, **45**, 246), and in cheese by the methods of Hostettler and of Erbacher (*Dtsch. MolkereiZtg*, 1928, **32**, 1074), are presented. The methods are critically examined. Mercurimetric determinations of Cl^- according to Hostettler, when compared with the argentimetric determinations of Drost and Erbacher, are more reliable and accurate in determining true values. A disadvantage is the time taken.
S.C.I. ABSTR.

5252. **Methods for the chemical analysis of butter.** British Standards Institution (2 Park Street, London, W.1). B.S. 769:1961. 22 pp.—Methods are specified for moisture, curd and salt and acidity, for volatile acids (Reichert, Polenske

and Kirschner values), iodine value, saponification value and refractive index of the fat, for pH of the butter serum and for the determination of Cu and Fe. N. E.

5253. **Detection of 2-alkoxyphenols in vanillin and ethylvanillin.** J. G. Hald (A/S Meco-Dumex, Anal. and Control Lab., Copenhagen). *Dansk Tidsskr. Farm.*, 1961, **35** (4), 73–77.—Traces of *o*-methoxyphenol in vanillin or of *o*-ethoxyphenol in ethylvanillin can be detected by the intense red coloration developed 20 min. after dissolution of the sample (0·05 g) in 74% H_2SO_4 (39 ml of water plus 61 ml of H_2SO_4) (3 ml). The colorations developed by the pure substances should not exceed that of a mixture of the $CoCl_2$ and $FeCl_3$ (1:19) U.S.P. colour standards. The smallest detectable amounts of the impurities (0·01%) correspond, respectively, with the least amount detectable by smell in vanillin and a distinctly detectable amount in ethylvanillin. Additions of vanitrope (6-ethoxy-*m*-propenylphenol) can similarly be detected by the same test. P. S. Arup

5254. **Determination of sorbitol in food products.** A. M. Manzone and C. J. Turi (Ist. Sup. di Sanità, Roma). *Ann. Chim., Roma*, 1961, **51** (3–4), 220–229.—The amount of sorbitol in a simple soln. is determined by measuring the optical rotation $[\alpha]_D^{25}$ of the soln. (> 2 g of sorbitol) to which has been added ammonium molybdate (4 g) and N-H_2SO_4 (25 ml) (to enhance the rotation) and made up to 100 ml, and comparing with a reference standard. In the presence of sugars that do not form complexes with ammonium molybdate, *e.g.*, glucose or sucrose, the $[\alpha]_D^{25}$ is first determined in aq. soln. and then in a soln. containing ammonium molybdate and H_2SO_4, and the sorbitol content is determined from the incremental increase in $[\alpha]_D^{25}$. The procedure with food products is indicated. L. A. O'Neill

5255. **Rapid method for the detection of sorbic acid in food.** A. Cesari, C. J. Turi and M. G. Malagodi (Ist. Sup. di Sanità, Roma). *Ann. Chim., Roma*, 1961, **51** (3–4), 312–317.—Sorbic acid is extracted from the foodstuff, *e.g.*, margarine, butter or cakes, by steam-distillation after acidification with H_3PO_4. The pH of the distillate is adjusted to 8 and the soln. is then oxidised with 0·1N-$KMnO_4$. In the presence of sorbic acid, acetaldehyde will be produced, and if the mixture is heated on a water bath in a vessel covered with a disc of filter-paper impregnated with Simon reagent (sodium nitroprusside and diethanolamine) a blue spot will be formed; 0·3 mg of sorbic acid may be detected. With wine, the sorbic acid must first be freed from ethanol by extraction and pptn. as Ba sorbate. L. A. O'Neill

5256. **Determination of the sulphur dioxide content of preserves.** K. Heintze (Bundesforschungsanst. f. Lebensmittelfrischhaltung, Karlsruhe, Germany). *Dtsch. LebensmittRdsch.*, 1961, **57** (3), 60–61.—The recovery of SO_2 from jam and marmalade by distillation methods is incomplete when H_3PO_4 is used for acidification. Residual SO_2 is generally higher in preserves made from dark-coloured fruits, probably owing to combination with the colouring matter of the fruit, and this bound SO_2 is completely liberated only by prolonged heating with a strong acid (*e.g.*, HCl) at a pH < 0·3. E. C. Apling

5257. **Chlorogenic acid in coffee beans and their alteration on roasting. III.** R. Krasemann (Stern-Apotheke, Hannover). *Arch. Pharm., Berlin*, 1961, **294** (5), 266–273.—Raw or roasted coffee (0·5 g) is extracted with 70% ethanol (10 ml) and the extract (0·1 ml) chromatographed on paper (S. & S. 2045a) with butanol - butyl acetate - acetic acid - water (9:47:28:16) as solvent. The separated chlorogenic acids, detected by their fluorescence in u.v. light, are eluted with ethanol (10 ml) and determined by measurement of the extinction at 324 mμ. A. G. Cooper

5258. **Recommended methods of analysis of barley, malt and adjuncts.** Institute of Brewing Analysis Committee (33 Clarges Street, London, W.1). *J. Inst. Brewing*, 1961, **67** (4), 315–327.—Apparatus and procedures are specified for sampling from various types of bulk containers and for the analysis of barley, pale malt, coloured malt (brown and crystal malts and roasted barley and malt) and unmalted grain (gelatinised grain, flaked maize, barley, oats and rice, and raw grain and grits). R. E. E.

5259. **Anthocyanogen content of barley, malt and beer. I. Methods of analysis.** W. O. S. Meredith (Grain Res. Lab., Winnipeg, Manitoba, Canada). *Proc. Amer. Soc. Brew. Chem.*, 1960, 48–50.—Two methods are described. In the first (Harris and Ricketts, *Anal. Abstr.*, 1960, **7**, 1929), the beer or extract of barley or malt is shaken with Nylon 66 and filtered. The Nylon residue is then dispersed in n-butanol - HCl (5:1) and heated in a boiling-water bath for 30 min. The Nylon dissolves, and the extinction of the coloured soln. is measured at 550 mμ. In the second method (*cf.* Wye and McFarlane, *Proc. 6th Congr. Eur. Brew. Conv., Copenhagen*, 1957, 299) the extract or beer is boiled with HCl for 30 min., cooled and washed with ethyl acetate, which is discarded. The aq. phase is then shaken with n-butanol, the org. layer is washed with dil. HCl, and its extinction is measured at 550 mμ. The extraction of colour was much greater by the Nylon procedure than by the Wye and McFarlane procedure. The results suggest that successive sparge liquors contain different relative proportions of two pigments. R. E. E.

5260. **Determination of gibberellins in malt.** L.-V. S. Lazer, W. E. Baumgartner, R. V. Dahlstrom and B. J. Morton (Abbot Lab., North Chicago, Ill.). *Proc. Amer. Soc. Brew. Chem.*, 1960, 85–89.—An isotope dilution procedure with gibberellic acid (**I**) labelled with ^3H (Baumgartner *et al.*, *J. Agric. Food Chem.*, 1959, **7**, 422) is described. For the determination of residual **I**, steeped barley was treated with labelled **I** at various concn., germinated and dried. The corns and rootlets were analysed separately by a complex purification procedure involving extraction with acetone containing a known amount of carrier **I**, followed by evaporation under reduced pressure, extraction with ethyl acetate and chromatography of the ethyl acetate residues on acid-washed alumina. Samples of **I** were then recovered by crystallisation and counted by a liquid-scintillation technique with an internal standard. For the determination of endogenous **I** in green malt, samples (2·5 to 5 kg) were extracted batchwise with aq. acetone containing labelled **I** at pH 2·5 and similarly purified, elution from the column being with 5% acetic acid in ethyl acetate. The fractions having the highest activity were combined, extracted with ethyl

acetate, again chromatographed and eluted with 3% acetic acid in ethyl acetate. The eluate was concentrated *in vacuo* and submitted to descending chromatography on paper with aq. NH_3 - butanol. The active spots were located by scanning, then cut out and extracted with ethanol. The extracts were treated with ^{14}C-labelled diazomethane and the methyl esters so obtained were chromatographed on paper with light petroleum - toluene - ethanol - water. The chromatograms were scanned, the areas corresponding to labelled **I** were cut out, extracted in the presence of the corresponding unlabelled ester, and the residues were recrystallised from light petroleum - ethyl acetate. The ^{14}C to ^{3}H ratios were determined by scintillation counting and compared with those for two standards carried through the procedure. Then endogenous **I** (μg) = $(R/r - 1) \times C$, where R and r are the ratios for sample and standard, respectively, and C is the amount (μg) of added labelled **I**. The results are discussed. R. E. E.

5261. **Determination of micro amounts of gibberellic acid in products of the brewing industry.** F. J. Holzer and N. R. Kuzel (Eli Lilly & Co., Indianapolis, Ind., U.S.A.). *Proc. Amer. Soc. Brew. Chem.*, 1960, 90–94.—The method depends upon measurement of the fluorescence of gibberellic acid in H_2SO_4 soln. *Procedure*—Ground malt (25 g) is mixed with phosphate buffer (pH 7) and dialysed twice at room temp., with shaking, against 100 ml of the same buffer. The combined dialysates are washed with ethyl acetate (2 × 50 ml), the aq. layer is adjusted to pH 2·0 with H_2SO_4 and washed with light petroleum (50 ml). The aq. phase is then extracted with isobutyl methyl ketone (3 × 50 ml) and the combined extracts are passed through a column containing a mixture of 25 g of $KHCO_3$ and 5 g of anhyd. Na_2SO_4. The column is then washed with ethyl ether (50 ml), dried by suction and eluted with methyl cyanide (50 ml). The eluate is evaporated to dryness in a current of air, and the residue is dissolved in water (10 ml). An aliquot (1 ml) is treated in an ice bath with 25 ml of chilled 85% H_2SO_4. After 1 hr. the fluorescence is read in a Klett - Summerson fluorimeter against a standard of 25 μg of gibberellic acid added to 25 g of control malt, and a blank of 25 g of control malt carried through the procedure. For beer, the sample (200 ml) is adjusted to pH 7 with the buffer soln., and the extraction and purification procedures are carried out as described for malt, omitting the dialysis step. Recoveries of 84 to 89% of gibberellic acid added to malt, and 75% of that added to beer, were achieved. The method can be used to determine amounts of 5 parts per 10^9 or less, and a single analysis takes 16 hr. R. E. E.

5262. **Problems in connection with the determination of bitter substances in hops and beer.** E. Mikschik (Brauerei Schwechat, A.-G., Wien, Austria). *Brauwissenschaft*, 1961, **14** (5–6), 272–278.—A review is presented in which the relative merits of the available methods of analysis and calculation are discussed with reference to outstanding problems. (39 references.) P. S. Arup

5263. **Analyses [of beer] simplified. I. Chlorides. II. Pasteurisation. III. Diacetyl.** J. L. Owades, J. Jakovac and C. Vigilante (Schwarz Lab., Inc., Mt. Vernon, N. York). *Proc. Amer. Soc. Brew. Chem.*, 1960, 63–67.—*Procedures*: (*i*) *For chlorides*—Titration with $HgNO_3$ soln., with diphenylcarbazone as indicator, is used. (*ii*) *Test for pasteurisation*—A test depending on the hydrolysis of sucrose by invertase in the beer is described. (*iii*) *For diacetyl*—De-gassed beer (100 ml) is distilled into a receiver containing 5 ml of water. When 15 ml of distillate has been collected the contents of the receiver are made up to 25 ml. An aliquot (5 ml) of this soln. is treated with 4% 1-naphthol soln. in isopropyl alcohol (1 ml) followed by a soln. of 0·3 g of creatine in 80 ml of 40% KOH soln. (0·5 ml), and the mixture is diluted to 10 ml and shaken for exactly 1 min. The extinction is then read after 5 to 6 min. against a reagent blank, and compared with a calibration curve (2·5 to 20 μg of diacetyl) prepared at the same time. A response is given by diacetyl and related compounds containing vicinal ketone groups. Recoveries of added diacetyl up to 1 p.p.m. ranged from 94 to 104%. R. E. E.

5264. **Measurement of beer turbidity in EBC formazin turbidity units.** W. Kleber and G. Franke (Wissenschaftl. Sta. f. Brauerei, Munich, Germany). *Brauwissenschaft*, 1961, **14** (5–6), 279–281.—To improve the accuracy of the measurement in the lower critical range, it is proposed to dilute the stock soln. 1:100 instead of 1:10 for comparative purposes. The results might with advantage be stated in the well-known Helm units as whole numbers. P. S. Arup

5265. **Determination of beer volatiles by gas chromatography.** A. P. Van der Kloot and F. A. Wilcox (Wahl-Henius Inst., Chicago, Ill., U.S.A.). *Proc. Amer. Soc. Brew. Chem.*, 1960, 113–116.—The apparatus previously described (*Ibid.*, 1959, 76) has been modified by removing the combustor and by introducing a helium purifier consisting of 25 ml of Molecular Sieve 5A. The detector is operated at 40° and the column at 60°, with a helium flow-rate of 200 ml per min. Two aliquots of the distillate from 200 ml of beer are passed through two columns (8 ft. × 0·25 in.), each containing Chromosorb (30 to 60 mesh) treated successively with conc. HCl, water, 0·1N-NaOH and water, and dried at 100°. The first column is prepared by dissolving 25 g of glycerol in methanol, slurrying it with 100 g of treated Chromosorb and drying *in vacuo*. The second column is prepared similarly, with 25 g of Carbowax 1500 in place of glycerol, and a 3-in. glycerol column is inserted between the Carbowax column and the detector. The first column serves for the determination of amyl acetate, isoamyl alcohol and 2-methylbutan-2-ol, and the second column for the determination of acetaldehyde, acetone, ethyl acetate, methanol, n-propanol and isobutyl alcohol. Identification is carried out with samples containing known amounts of the compoments, in aq. ethanol, carried through the distillation procedure. Results for the frequency distributions of methanol, n-propanol and isobutyl alcohol in 43 American beers are presented as histograms. R. E. E.

5266. **Method for the detection of chill-proofer protease in beer.** P. R. Glenister and M. Burger (J. E. Siebel Sons' Co., Inc., Chicago, Ill., U.S.A.). *Proc. Amer. Soc. Brew. Chem.*, 1960, 117–122.—The method of Gates (*Proc. Soc. Exp. Biol. Med.*, 1926–27, **24**, 936) is modified. Camera roll-film (Kodak Verichrome Pan-120) is cut into pieces ≃2 cm × 2 cm. The sample of beer (≃600 ml) is divided into two portions, one of which (test)

is transferred to a 250-ml conical flask so as to fill it to the brim, and the second (control) is boiled for 5 min. under reflux, cooled, and similarly transferred to a 250-ml flask. Both flasks are then warmed to 40° in a water bath, and a piece of the cut film is completely immersed vertically in each. The digestion is allowed to proceed at 40°; the gelatin emulsion is gradually dissolved from the film in the test flask, and the test is stopped as soon as the difference between the two pieces of film is evident. If no difference appears in 60 min. the result is regarded as negative. R. E. E.

5267. **Method for the determination of dissolved oxygen and its application to the brewing process.** P. Jenkinson and J. Compton (John Labatt, Ltd., London, Ontario, Canada). *Proc. Amer. Soc. Brew. Chem.*, 1960, 73–77.—The blue colour produced on the addition of a soln. of reduced indigo carmine is measured and compared with that of standards. Details are given for carrying out the determination on beer and wort. R. E. E.

5268. **Determination of oxidisable substances in beer.** J. L. Owades and F. Zientara (Schwarz Lab., Inc., Mt. Vernon, N. York). *Proc. Amer. Soc. Brew. Chem.*, 1960, 68–71.—The method depends on the reduction of the colour of α,α-diphenyl-β-picrylhydrazyl (**I**) (prep. described) within a specified time. Two classes of compound can thus be determined, one reacting rapidly (within 5 min.) and the other more slowly. *Procedure*—To 8 ml of solvent [2-methoxyethanol - water (1:1) containing 8% of KCl] add 0·2 ml of **I** soln. and 0·2 ml of de-gassed beer. Make up to 10 ml with solvent and measure the extinctions at 520 mμ against a solvent blank after 5, 10 and 30 min. Also measure a reagent blank at the same time-intervals at least once daily, and correct for fading of the **I** soln. The concn. of oxidisable substances at time t (H_t) in p.p.m. of hydrogen is given by $H_t = \Delta A/A_s \times 1250 \times C$, where ΔA is the difference between the extinctions of the test soln. and the **I** blank at time t, A_s is the extinction of the diluted dye (0·25 ml of **I** soln. diluted to 10 ml with ethanol) at zero time and C is the concn. of the **I** soln. in moles per litre. The standard deviation is 0·01 p.p.m. (100 measurements). Simple mercapto-compounds (*e.g.*, cysteine) and reductones (*e.g.*, ascorbic acid) react rapidly, while glutathione, beer tannins and melanoidins react more slowly. Results are quoted for 21 commercial beers. R. E. E.

5269. **Colorimetric procedure for the simultaneous determination of tartaric and lactic acids in wine and must.** H. Rebelein (Staatl. Chem. Untersuchsanst., Würzburg, Germany). *Dtsch. LebensmittRdsch.*, 1961, **57** (2), 36–41.—*Procedure*—Fill a column (25 mm × 10 to 11 mm diam.) with strongly basic ion-exchange resin (Merck III) in the acetate form. Wash the column with 0·5% acetic acid (4 × 10 ml). Add the sample of wine or must (10 ml), and wash the resin with water (7 × 10 ml), controlling the flow rate at from 1 to 1·5 drops per sec. Elute the acids with 0·5M-Na_2SO_4, collecting 100 ml at a flow rate of from 2 to 3 drops per sec. *For tartaric acid*—To a portion of the eluate (50 ml) add 2% ammonium vanadate soln. (5 ml). Add, with mixing, 2N-H_2SO_4 (2 ml) and measure the extinction at 530 mμ after exactly 1·5 min. Calculate the crude tartaric acid from a standard curve, and correct the result by means of the formula, true tartaric acid (g per litre) $= R + (G - R) \times 0{\cdot}055$, where G = total acidity of the sample, calculated as tartaric acid. *For lactic acid*—To a second portion of eluate, or a dilution in 0·5M-Na_2SO_4 (10 ml), add 0·1N-cerium sulphate (0·7N in H_2SO_4) (10 ml). Heat at 65° for 10 min., cool to 20°, add 2·5N-NaOH (5 ml), mix and filter. After exactly 5 min. measure the filtrate (15 ml) into a 50-ml cylinder containing 27% Na acetate soln. (5 ml) and 2N-H_2SO_4 (2 ml). Add a 2% soln. of sodium nitroprusside (5 ml) and set aside for 2 min. Add a 10% soln. of piperidine (5 ml), mix, and measure the extinction at 570 mμ or 530 mμ. Recoveries of added tartaric and lactic acids from wine are very close to 100%. E. C. Apling

5270. **Determination of chloropicrin in wine.** V. Astegiano and M. Malta (Stazione Enologica Sperimentale, Asti, Italy). *Chim. e Ind.*, 1961, **43** (3), 266–270.—The chloropicrin in a 500-ml sample of wine is extracted with xylene (5 ml) and recovered from the extract by steam-distillation. A 1-ml aliquot of distillate is heated with dimethylaniline (1 ml) and a drop of 30% H_2O_2 soln. for 15 min. at 110°, when a yellow - red colour appears in the presence of chloropicrin. With benzoyl peroxide a yellow - red colour develops after 10 min. in the cold in the presence of chloropicrin (>150 μg per litre), and is measured absorptiometrically at 350 mμ. C. A. Finch

5271. **Determination of the acid values of dark oils by means of the chemiluminescent indicator lucigenin [5,5′-biacridyl dimethonitrate].** E. Ya. Yarovenko and G. N. Kosheleva. *Zavod. Lab.*, 1961, **27** (4), 407–408.—The sample (5 to 10 g) is dissolved in a mixture of 40 ml of benzene and 20 ml of methanol, and 3 drops of 15% H_2O_2 (obtained by dilution of 30% H_2O_2 with methanol) and 0·5 ml of a 0·1% soln. of lucigenin in alcohol are added. Titration is carried out with 0·05N-Na methoxide in the dark. G. S. Smith

5272. **Study of unsaponifiable matter by chromatography on silicic acid.** P. Capella, G. de Zotti, G. S. Ricca, A. F. Valentini and G. Jacini (Staz. Speriment Olii Grassi, Milan). *Riv. Ital. Sostanze Grasse*, 1961, **38** (3), 198–202.—The unsaponifiable matter of fatty oils freed from all fatty acid or soap is dissolved in hexane, adsorbed on a silicic acid column and eluted with a series of solvents of increasing polarity. The components are separated into their main groups, *viz*, hydrocarbons, waxes, sterol esters, alcohols and sterols, which are examined by colour tests and i.r. spectrophotometry. The results obtained with an extracted olive oil are given; the principal sterol is β-sitosterol. L. A. O'Neill

5273. **Chromatographic analysis of fatty substances in the vapour phase. II. Technique of operation and chromatograms of some animal and vegetable oils and fats.** S. Anselmi, L. Boniforti and R. Monacelli (Ist. Sup. Sanità, Roma, Italy). *R.C. Ist. Sup. Sanit.*, 1960, **23** (12), 1016–1036.—Characteristic gas chromatograms are given for the fatty acids in authentic natural fatty oils, with particular reference to olive oils of various origins and of oils commonly used for the adulteration of olive oils. For chromatography, the methyl esters of the fatty acids are prepared by boiling the oils in methanolic soln. with Na methoxide and extracting the methyl esters with ether. The chromatographic column of Celite C22 contains 25%

of the polyester of succinic acid with ethanediol and is run at 200°. Quant. determination of the fatty acids is effected by measuring the areas under the respective peaks in the chromatogram. These areas are multiplied by correction factors dependent on the number of C atoms in the fatty acid molecule. Correction factors for the methyl esters of octanoic, decanoic, lauric, myristic, palmitic, stearic, arachidic and behenic acids are given. Chromatograms are given for various natural (pressed) and rectified (solvent extracted) oils.

J. I. M. JONES

5274. **Spectrophotometric differentiation of expressed and extracted cacao butter.** E. Lauber (Eidg. Gesundheitsamt, Bern, Switzerland). *Mitt. Lebensmitt. Hyg., Bern*, 1961, **52** (2), 116–122.—By measurement of the u.v. extinction of a 0·5% soln. in heptane in a 1-cm cell at 270 mμ, expressed and extracted cacao butter can be differentiated, and additions of more than 10% of the latter to the former can be detected. R. CLARKE

5275. **Analysis of lipids and oxidation products by partition chromatography. Dimeric and polymeric products.** E. N. Frankel, C. D. Evans, H. A. Moser, D. G. McConnell and J. C. Cowan (N. Reg. Res. Lab., Peoria, Ill., U.S.A.). *J. Amer. Oil Chem. Soc.*, 1961, **38** (3), 130–134.—A liquid-partition chromatographic method was developed to determine dimers in fats. Silicic acid treated with 20% methanol in benzene served as the stationary phase, and 2% methanol in benzene as the mobile phase. Chromatographic separation of free fatty acids from oxidised deodorised oils gave 3 well-separated fractions—(*i*) unoxidised acids, (*ii*) dimeric or polymeric acids, and (*iii*) a polar fraction eluted with ethyl ether. A 96 to 100% recovery of acidic materials from the column was obtained with good reproducibility. There was a linear relationship between dimer content of deodorised soya-bean oil and the peroxide value of the oil before deodorisation, with a standard deviation about the regression line of $\pm 0{\cdot}264$. An increase of 1% in dimer concn. corresponds to an increase in peroxide value of $\simeq 40$. The dimer content of different vegetable oils varied from 1 to 3%. The method can be used to determine the degree of oxidation of an oil before deodorisation, and also to follow various phases of fat oxidation, polymerisation and processing.

E. S. LANE

5276. **Analyses of lipids and oxidation products by partition chromatography. Fatty acid hydroperoxides.** E. N. Frankel, C. D. Evans, D. G. McConnell and E. P. Jones (N. Reg. Res. Lab., Peoria, Ill., U.S.A.). *J. Amer. Oil Chem. Soc.*, 1961, **38** (3), 134–137.—A liquid-partition chromatographic method was developed for the isolation and determination of hydroperoxides in autoxidised fatty acids and their methyl esters. The use of 2 to 4% methanol in benzene as mobile solvent separated the hydroperoxides from unoxidised fatty acids or methyl esters and from secondary and polymeric decomposition products. In the analyses of oxidised fatty acids, ethyl ether was necessary to elute the secondary decomposition products. Saponification of autoxidised fatty esters destroyed the peroxides as determined iodimetrically, but the resulting acids contained a fraction that was eluted in the same position as hydroperoxide acids and is believed to be a monomeric hydroxy-fatty acid containing conjugated *cis-trans* and *trans-trans* unsaturation. Fatty ester hydroperoxides were isolated chromatographically in yield and purity comparable with those reported for counter-current distribution methods. The concn. of methyl linoleate hydroperoxide determined chromatographically were smaller than indicated by the peroxide value and diene conjugation of the autoxidised methyl linoleate. E. S. LANE

5277. **Characterisation of the methyl esters of higher saturated fatty acids by means of infra-red spectroscopy and gas chromatography.** U. Pallotta (Bologna Univ., Italy). *Riv. Ital. Sostanze Grasse*, 1961, **38** (3), 191–197.—The normal saturated even-numbered C_{18} to C_{26} fatty acids may be differentiated by means of the i.r. spectra of their methyl esters, determined in a liquid paraffin mull, particularly from the number of bands in the 7·5 to 8·5-μ region, which is equal to half the number of C atoms in the chain. The C_{20} to C_{26} acids also differ from stearic acid by having a third band in the 8·26 to 8·5-μ region. Some identification is possible of binary mixtures, but with more complex mixtures a preliminary fractional crystallisation is necessary to reduce the components to $\not> 2$. The use of the technique for examining adulteration of olive oils is described. The gas-chromatographic separation of the methyl esters of the C_{12} to C_{24} acids is reported. L. A. O'NEILL

5278. **The determination of thiamine by the thiochrome method.** H. Haugen (Medical Dept. A, Rikshospitalet, Oslo, Norway). *Scand. J. Clin. Lab. Invest.*, 1960, **12** (3), 253–260.—A re-investigation of the thiochrome method is reported. A slow addition of the thiamine soln. to the pre-mixed soln. of NaOH and $K_3Fe(CN)_6$ is recommended. The isobutyl alcohol and aq. phases should be separated immediately after centrifugation. The temp. of both the sample and the quinine standard should be kept constant during the fluorimetric reading. Excess of ferricyanide is harmful. The oxidation time should be carefully controlled. P. PEDERSEN

See also Abstracts—**4918**, Vitamin C in foods. **5047**, Methoxy-groups in pectin. **5074**, Organotin compounds in milk. **5131**, Tin in foods. **5167**, Detection of diglycerides. **5168**, Determination of triglycerides. **5170**, Separation of lipids on silicic acid. **5176**, Thiamine in urine. **5212**, Oestrogens in milk. **5242**, Chloramphenicol in crab meat. **5283**, Amylase in malt and barley. **5310**, Determination of ^{14}C-labelled fatty acids.

Sanitation

Analysis of air, water, sewage, industrial wastes, industrial poisons.

5279. **Determination of manganese, chromium and iron in air by a polarographic method.** M. S. Bŷkhovskaya and I. A. Orlova (Inst. of Work Hygiene and Occupational Diseases). *Zavod. Lab.*, 1961, **27** (5), 540–542.—Air at the rate of 8 to 10 litres per min. is passed through a perchlorovinyl-resin filter, which is then ashed and fused at 900° with 0·5 g of a molten mixture of Na_2CO_3 and KNO_3 (2:1). The cooled residue is heated with 5 ml of water and 1 to 2 drops of ethanol. The soln. is separated from the insol. matter by means of a centrifuge and evaporated, the residue

is dissolved in 2 ml of 0·5N-HCl, and the soln. is mixed with 2 ml of 0·5N-triethanolamine, 2 ml of 20% NaOH soln., 0·5 g of Na_2SO_3 and two drops of 0·5% gelatin soln. After 10 min. the polarographic wave for CrO_4^{2-} is recorded. The insol. matter is dissolved in 2 to 3 ml of aq. HCl (1:1) and the soln. is evaporated to dryness. Water (2 ml) is added and the evaporation is repeated. The residue is dissolved in 2 ml of 0·5N-HCl and the soln. is mixed with 2 ml of 0·5N-triethanolamine, 2 ml of 20% NaOH soln., 0·5 g of Na_2SO_3 and 2 drops of 0·5% gelatin soln. After 10 min. the polarogram for Mn and Fe is recorded.

G. S. SMITH

5280. **Methods of testing water used in industry.** British Standards Institution (2 Park Street, London, W.1). B.S. 2690: 1956. Addendum No. 1, 1960. Amendment No. 1 (20.7.61). 2 pp.—A new diagram of the apparatus for the determination of CO_2 and modifications of the equations in the method for the determination of dissolved oxygen are given. N. E.

5281. **Compensation for temperature variations within the cell of a dissolved-oxygen indicator/recorder incorporating a wide-bore dropping-mercury electrode.** A. Littlewood (D.S.I.R., Warren Spring Lab., Gunnels Wood Rd., Stevenage, Herts., England). *Analyst*, 1961, **86**, 359–360.—In the polarographic determination of O dissolved in water (Briggs *et al.*, *Ibid.*, 1958, **83**, 304), a dropping-mercury electrode is maintained at a constant negative potential against a zinc electrode in a cell through which the water flows at a constant rate, the cell current being then a measure of the concn. of O in the water. To compensate for variations in current produced in the measuring cell by changes of temp., a batch of thermistors is incorporated in the cell. Hitherto the values of the shunt and series resistances have been determined experimentally. It has now been found possible to calculate the values of these, and the mathematical basis of the calculation is given.

A. O. JONES

5282. **Comparison of chlorine-determination methods in waste waters.** M. C. Rand and J. V. Hunter (Syracuse Univ., N.Y.). *J. Wat. Pollut. Control Fed.*, 1961, **33** (4), 393–398.—The results and reproducibility of modifications of the *o*-tolidine method, the bacteriological chlorine-requirement test and the amperometric method were compared. The first two methods gave results in good agreement. The third method gave lower and inconsistent results.

O. M. WHITTON

5283. **Determination of chlorine dioxide in water.** A. T. Palin. *Proc. Soc. Wat. Treatm. Exam.*, 1960, **9**, 81.—The *p*-amino-*NN*-diethylaniline (**I**) method for the determination of Cl and chloroamines (*Anal. Abstr.*, 1959, **6**, 3793) can be used to determine ClO_2 if the sample is first acidified in the presence of KI and the pH value is then adjusted to 6·0 to 6·5 by addition of $NaHCO_3$; subsequent addition of **I** soln. gives full colour formation corresponding to the total available Cl content of the ClO_2. By using both this method and the normal $(NH_4)_2SO_4.FeSO_4$ procedure it is possible to analyse a mixture of ClO_2, Cl and chloroamines. WAT. POLLUT. ABSTR.

5284. **Determination of monomeric silica and its primary polymers in water, spectrophotometrically as β-silicomolybdic acid, in the presence of interfering ions.** B. Visintin and S. Monteriolo (Ist. Sup. di Sanità, Roma). *Ann. Chim., Roma*, 1961, **51** (3–4), 266–287.—Conditions for the determination of monomeric and dimeric silica in water have been examined and the following procedure is proposed. To 50 ml of the sample (containing 0·2 to 40 mg of SiO_2 per litre) are added 1 ml of 4·5N-HCl and 2 ml of 10% ammonium molybdate soln. After 3 min. the extinction at 400 mμ is measured spectrophotometrically, from which the concn. of SiO_2 can be calculated. If phosphate is present, 1 ml of 15% mannitol soln. is added before the spectrophotometric reading. Fluoride is eliminated with Al^{3+} or H_2BO_3, and if Fe^{3+} are present in concn. >1 mg per litre, the sample is diluted. Other ions likely to be found do not interfere up to certain limits. L. A. O'NEILL

5285. **Analysis of phenolic disinfectants.** D. E. Dean, R. Suffis and A. Levy (Shulton Inc., Clifton, N.J., U.S.A.). *Soap, N.Y.*, 1961, **37** (3), 87, 89, 101.—Potentiometric titrations and direct absorption spectrophotometry lack sensitivity and/or specificity; this disadvantage is largely overcome by making use of the dependence of u.v. spectra of the phenols and the aromatic amines on pH. At selected pH values, the spectral differences are sufficiently marked to enable a useful analysis to be made. The behaviour of various substituted phenol derivatives is discussed, and u.v. absorption spectra for hexachlorophane, salicylanilide, *p*-phenylenediamine and nitro-*p*-phenylenediamine in the appropriate pH ranges are shown.

S.C.I. ABSTR.

See also Abstracts—**5004**, Uranium in natural waters. **5005**, Uranium in atmospheric dusts. **5060**, Acraldehyde in air. **5077**, Phenols in industrial wastes.

Agricultural analysis

Soil, fertilisers, herbicides, pesticides, animal feeding-stuffs.

5286. **Volumetric determination of calcium in phosphate and plant ash.** C. Sørensen (Royal Vet. and Agric. Coll., Copenhagen, Denmark). *Acta Agric. Scand.*, 1960, **10** (4), 301–311.—Adsorption of phosphate on a strong anion-exchange resin (Dowex 2) in acetate form was found to be most convenient. The percolate could be used directly for the EDTA titration of Ca. The influence of the salt concn. in the samples was investigated. P. PEDERSEN

5287. **Methods for the assessment of beet quality. II. Determination of particular non-sugars in beet.** A. Carruthers and J. F. T. Oldfield (British Sugar Corporation Ltd., Bramcote, Nottingham). *Int. Sugar J.*, 1961, **63**, 103–105.—It is considered that determinations of individual non-sugars provide more useful criteria for the assessment of beet quality than the traditional methods. Methods are given for these non-sugars. For cations, Na and K are determined by flame photometry, and Mg and Ca by EDTA titration. Betaine and pyrrolidonecarboxylic acid are separated by ion-exchange resin and determined colorimetrically with ninhydrin. Amino-nitrogen is determined by alkaline distillation of NH_3 and titration. Anions

are determined by treatment with a strong cation-exchange resin, followed by titration of total acids. Invert sugar is determined by 2,3,5-triphenyltetrazolium chloride (*Anal. Abstr.*, 1956, **3**, 234).
R. CLARKE

5288. **Rapid method for the determination of amylase activity in malt and barley extract in breeding trials.** D. Lau (Forschungsstelle f. Getreidezüchtung, Kloster Hadmersleben, Germany). *Brauwissenschaft*, 1961, **14** (4), 183–187.—The substrate is prepared by dissolving soluble starch (1 g) in acetate buffer (pH 4·3) (100 ml). Finely ground malt (5 g) is extracted with 200 ml of water for 5 min. at 20°; the mixture is filtered and 2 ml of the filtrate is diluted with 20 ml of water. Finely ground barley is extracted for 10 min. at 40° with 50 ml of citrate buffer (pH 5·2) and 50 ml of water to which 5 ml of 2% cysteine hydrochloride soln. has been added. The mixture is filtered and the filtrate is used for the determination. For the determination of amylase activity, 1 ml of the enzyme soln. is added to 1 ml of substrate and maintained for 15 min. at 20°; 2 ml of aq. 3,5-dinitrosalicylic acid soln. (prep. described) is then added and the mixture is heated for 5 min. in a boiling-water bath. After cooling the soln. and adding 20 ml of water, the extinction is measured in a Pulfrich photometer with filter S53. A calibration curve is prepared from maltose soln. The results agree well with those by conventional methods.
J. V. RUSSO

5289. **Crude fibre analysis. II. Comparison of different methods for determining crude fibre.** E. Mergenthaler (Dtsch. Forschungsanst. f. Lebensmittelchem., München, Germany). *Z. Lebensmitt-Untersuch.*, 1961, **114** (1), 18–26.—Analyses are given of 52 different types of plant material by the following methods—the original standard method with 1·25% H_2SO_4 and 1·25% KOH, the methods of Fincke and Thaler (*Ibid.*, 1942, **84**, 97), Hampel (*Ibid.*, 1950, **91**, 1), König (*Ibid.*, 1898, **1**, 3; 1903, **6**, 770), and Glimm and Hansen (*Vorratspfl. u. LebensmittelForsch.*, 1940, **3**, 89) and the ethanediol method. The results show considerable variations. None of the later methods shows any particular advantage with respect to reproducibility or correlation with the original standard method. The Fincke and Thaler, Glimm and Hansen and the ethanediol methods are especially convenient for routine analyses. The various procedures are described.
P. S. ARUP

5290. **Studies in mineralisation of sulphur from soil organic matter.** N. J. Barrow (Div. of Plant Ind., C.S.I.R.O., Pastoral Res. Lab., Armidale, N.S.W.). *Aust. J. Agric. Res.*, 1961, **12** (2), 306–319.—In the determination of available S by pptn. as $BaSO_4$, extraction of soil samples with a 0·15% soln. of $CaCl_2$ instead of with water improves the correlation between chemical and biological values. The $CaCl_2$ soln. gave complete extraction of added SO_4^{2-}, but extracted less total SO_4^{2-} than did water.
E. C. APLING

5291. **Identification of insecticides by thin-layer chromatography.** J. Bäumler and S. Rippstein, (Forensic Institute, Univ. Basel). *Helv. Chim. Acta*, 1961, **44** (4), 1162–1164 (in German).—Thin-layer chromatography was applied to the separation of some insecticides, *viz*, phosphorothioic esters and chlorinated pesticides. For identification of the phosphorothioic esters a solution of $PdCl_2$ was used. The spot-test reagent for the chlorinated compounds (*e.g.*, DDT and hexachlorocyclohexane), was *p*-amino-*NN*-dimethylaniline.
M. SULZBACHER

5292. **Recommended analytical methods for pesticides. I. Mercury in formulated or technical products.** Collaborative Pesticides Analytical Committee (Plant Pathol. Lab., Harpenden, Herts., England). *FAO Plant Protection Bull.*, 1960, **9** (2), 19–28.—Three basic methods, HG1, HG2 and HG3, together with minor modifications, are described, and recommendations are made with respect to the method best suited for the determination of Hg in any one of a comprehensive range of such products. In method HG1, recommended for use when lead arsenate or Cu is present, the sample is heated with CaO and sucrose, and the distilled Hg, after dissolution in HNO_3, is titrated with NH_4SCN. In method HG2, which is recommended for most technical products, the sample is decomposed with conc. H_2SO_4 and KNO_3, with subsequent titration of Hg with thiocyanate. When large amounts of $CaCO_3$ or highly chlorinated compounds are present, the Hg is first isolated as HgS, before dissolution in strong acid. Method HG3 and its modifications, which provide alternative procedures to those described under HG2, are based on the decomposition of the compound with glacial acetic acid and zinc wool, dissolution of the separated amalgam in HNO_3 and subsequent volumetric determination of Hg with thiocyanate. This method is specially recommended for use with compounds having a high fat content.
I. A. SIMPSON

5293. **CIPC [chlorpropham, isopropyl 3-chlorophenylcarbamate] residues in vegetables.** H. J. Hardon, H. Brunink and A. F. H. Besemer (Keuringsdienst van Waren, Amsterdam, Netherlands). *Dtsch. LebensmittRdsch.*, 1961, **57** (2), 31–35.—The extraction and colorimetric determination of CIPC residues is described. *Procedure*—Wash the plant material (≃1 kg) with water and reject the outer portions. Set aside in light petroleum for 3 days, evaporate an aliquot of the extract and heat with a mixture of acetic, hydrochloric and phosphoric acids (15 ml) (*cf.* Bissinger and Friedenburg, *J. Ass. Off. Agric. Chem.*, 1951, **34**, 812). Make alkaline and steam-distil, collecting 40 ml. To the distillate add 4N-HCl (2 ml) and a 0·25% soln. of $NaNO_2$ (1 ml). After 10 min., add a 2·5% soln. of ammonium sulphamate (1 ml) and, after a further 10 min., a 1% soln. of *N*-1-naphthylethylenediamine hydrochloride (1 ml). Measure the extinction at 350 mμ and evaluate from a standard curve.
E. C. APLING

5294. **Colorimetric determination of amprolium.** E. P. Schulz and C. R. Szalkowski (Merck & Co., Inc., Rahway, N.J., U.S.A.). *Anal. Chem.*, 1961, **33** (6), 795–798.—Amprolium [1-(4-amino-2-n-propyl-5-pyrimidinylmethyl)-2-picolinium chloride hydrochloride] reacts with napthalene-2,7-diol, $K_3Fe(CN)_6$ and KCN in alkaline methanol to produce a chromogen, which is measured at 530 mμ. The method appears to be specific for amprolium and its homologues, and is reproducible to ±0·7%.
G. P. COOK

See also Abstracts—**4918**, Nitrofurazone and furoxone in feeding-stuffs. **4953**, Hg in soil. **4990**, Phosphorus in fertilisers. **5074**, Organotin compounds in plants. **5130**, Trace metals in plants. **5140**, *p*-Dianilinobenzene in animal tissues.

5.—GENERAL TECHNIQUE AND APPARATUS

General

5295. **A simple and accurate micro-burette.** I. Holm-Jensen (Pharmacol. Inst., Univ., Aarhus, Denmark). *Scand. J. Clin. Lab. Invest.*, 1960, **12** (2), 247–248.—The design is based on the principle of controlling the delivery by means of the surface tension of the titration fluid in the tip of the burette. P. PEDERSEN

5296. **Micro-apparatus for extraction and distillation.** J. Erdos (Org. Chem. Res. Lab., Tech. Coll., Mexico D.F.). *Mikrochim. Acta*, 1961, (3), 515–516 (in German).—A simple and cheap apparatus for liquid - solid extraction, and for the subsequent removal of the solvent, is described. R. M. ROWLEY

5297. **Extraction in liquid - liquid systems with pulsing jets.** L. Massimilla and G. Volpicelli (Naples Univ., Italy). *Ric. Sci.*, 1960, **30** (12, Suppl.), 2458–2466.—The rates of extraction of acetic acid from soln. in benzene, toluene, *o*-xylene, n-heptane and isoamyl alcohol by water, by means of pulsing jets and with water as the dispersing phase, have been measured for various jet sizes and frequencies and intensities of pulsation, and compared with results given by continuous jets. L. A. O'NEILL

5298. **Laboratory film-evaporator for rapid concentration of dilute solutions [in organic solvents].** M. T. Bush (Dept. of Pharmacol., Vanderbilt Univ. Sch. of Med., Nashville, Tenn., U.S.A.). *Anal. Biochem.*, 1960, **1** (3), 274–277.—Apparatus suitable for concentrating large volumes of soln. in volatile organic solvents is described. The soln. is passed down a spiral of tantalum wire or 1-mm glass rod on the inside of a heated column, and the concentrate is collected in a reservoir at the base. Provision is made for recovery of the solvent, and vacuum can be applied. The apparatus is not suitable for aq. soln. J. R. HUDSON

5299. **Recovery of chloroform after use in solvent-extraction analyses.** H. Green (B.C.I.R.A. Lab., Alvechurch, Birmingham). *B.C.I.R.A. Journal*, 1961, **9** (3), 382–384.—The cost and difficulty of disposal of $CHCl_3$ after use as an extracting medium with 8-hydroxyquinoline, diethyldithiocarbamate and cupferron makes recovery essential, and methods for reclamation and analysis are given. No significant difference was found between results of tests made with recovered $CHCl_3$ and that newly purchased. Cupferron residues are dangerous to treat and should be disposed of after excess of $CHCl_3$ is poured off; 8-hydroxyquinoline residues can be distilled directly, but diethyldithiocarbamate soln. require special treatment (method described). C. H. COWPER-COLES

5300. **Applications of a new gel filtration method for molecular separation.** A. Bill, N. Marsden and H. Ulfendahl (Inst. of Physiology, Univ. Uppsala, Sweden). *Scand. J. Clin. Lab. Invest.*, 1960, **12** (4), 392–395.—Some experiments with polydextran gel ("Sephadex") filtration in the separation of free iodide from ^{131}I-containing albumin, radioactive Na_2CrO_4 from haemoglobin, and glucose from dextran are reported and discussed. The method is very simple and good separations can be rapidly obtained. P. PEDERSEN

5301. **Simple and convenient apparatus for generating carbon dioxide of the highest purity for nitrogen determinations by the Dumas - Pregl method.** M. Hochenegger (Forschungsabt., Mikroanal. Lab., E. Merck. A.-G., Darmstadt, Germany). *Mikrochim. Acta*, 1961, (3), 431–435 (in German).—An apparatus for the preparation of air-free CO_2 from $KHCO_3$ and H_2SO_4 is described. The following advantages are claimed—(*a*) no CO_2 is wasted; (*b*) mercury cannot be splashed into the gas stream; (*c*) both filling and emptying of the apparatus are simple; (*d*) specially pure reagents are unnecessary; (*e*) the apparatus is simple to make; and (*f*) the apparatus is always ready for use. R. M. ROWLEY

5302. **pH standard for blood and other physiological media.** V. E. Bower, M. Paabo and R. G. Bates (Nat. Bureau of Standards, Washington, D.C., U.S.A.). *Clin. Chem.*, 1961, **7** (3), 292–293.—A new pH standard to facilitate measurement of the pH of blood and other fluids in the range 7 to 8 is announced. It is prepared by dissolving 1·179 g of KH_2PO_4 (0·008695M) and 4·303 g of Na_2HPO_4 (0·03043M) in pure NH_3-free H_2O and diluting to 1 litre at 25°; $\mu = 0{\cdot}1$. The pH values of this soln. are listed at temp. from 0° to 50°, *e.g.*, at 15°, 7·450; at 20°, 7·430; at 25°, 7·413; and at 38°, 7·382. H. F. W. KIRKPATRICK

5303. **Thermogravimetric analysis.** G. M. Lukaszewski and J. P. Redfern (Battersea Coll. of Technol., London). *Lab. Practice.*—**I. Introduction,** 1961, **10** (7), 469–473. **II. Applications,** 1961, **10** (8), 552–555. **III. Techniques used in conjunction with thermogravimetry,** 1961, **10** (9), 630–632.

5304. **Determination of the evaporation curve for liquids by the thermogravimetric method.** F. Paulik, J. Paulik and L. Erdey. Brit. Pat. 865,074, date appl. 25.9.57.—The method enables the evaporation testing of liquids to be carried out quickly and accurately with as little as 0·5 to 1 g of liquid. The measurement departs from known practice by not determining the distillate vol., but by continuously determining the wt. of liquid residue by means of a thermobalance and by determining the temp. of the escaping vapour by means of a thermocouple. J. M. JACOBS

Chromatography, ion exchange, electrophoresis

5305. **Efficiency parameters in linear chromatography and their use for analytical purposes.** P. C. Haarhoff and V. Pretorius (Dept. of Phys. Chem., Univ., Pretoria). *J. S. Afr. Chem. Inst.*, 1961, **14** (1), 22–42.—A development of efficiency parameters is given that is more rigorous than that of Purnell (*J. Chem. Soc.*, 1960, 1268), and is applicable to all linear chromatographic processes. With an approach to the problem of the over-all efficiency of the separation of a given pair of solutes via the impurity-factor treatment developed by Glueckauf (*Trans. Faraday Soc.*, 1955, **51**, 34) it is shown that that part of the over-all efficiency due to the column efficiency should be measured by the parameter $k^3N/(1 + k)^3$, for which the

term "number of theoretical plates based on apparent retention" is proposed, and in which N is the number of theoretical plates and k is the ratio at equilibrium of the mass of solute in the stationary phase to the mass in the mobile phase. A definition of a measure of column efficiency that is independent of the column length is also proposed. The use of efficiency parameters in analytical chromatography is also discussed, with particular reference to the values of these parameters which are required to separate two solutes to a given extent. E. G. CUMMINS

5306. **Improvements in chromatography tanks.** Shandon Scientific Co., Ltd. [Inventor: E. R. Shandon]. Brit. Pat. 873,880, date appl., 2.4.59.—The cylindrical tank is shaped internally at the upper end to enable the glass solvent-trough to be sealed therein without the aid of additional support fittings. J. M. JACOBS

5307. **Improvements in or relating to frames for use in two-dimensional paper chromatography.** Vitamins, Ltd. [Inventors: J. Green and P. R. Wyatt]. Brit. Pat. 872,905, date appl. 19.3.59.—The frame comprises two parallel end-plates secured together with paper-supporting bars extending perpendicularly between them and provided with transverse slots to receive paper sheets and to space the sheets apart. The paper sheets are secured to bores in the bars by means of skewers.

J. M. JACOBS

5308. **The elution of paper chromatograms.** J. R. Broich (Dept. of Surgery, Albert Einstein Coll. Med., Yeshiva Univ., New York). *J. Chromatography*, 1961, **5** (4), 365–366 (in English).—The apparatus consists of a 5-cm length of thick-walled capillary (0·75 mm bore) fused to a length of 1-cm tubing and held inside a test-tube (15 cm × 2 cm) by means of a paper-clip with the lower 5 mm of the clip bent at right angles. The paper is folded and placed inside the eluter. With the given sizes, ethanol (2 ml) drains in 4 min. Dehydroepiandrosterone (3β-hydroxyandrost-5-en-17-one) (10 to 50 μg) was chromatographed on Whatman No. 1 paper and the spots were eluted with 2-ml portions of ethanol or methanol to a total vol. of 10 ml. The recovery averaged 94 $\pm$3·5%.

G. RUSSELL

5309. **A simple apparatus for rapid drying of paper-strip chromatograms.** J. Gołdyn (Jagiellonian Univ., Kraków, Poland). *J. Chromatography*, 1961, **5** (4), 372–373 (in English).—The device is intended to facilitate drying at room temp. It consists of a glass tube (6 cm × 30 cm) attached to glass Buchner funnels (6 cm bottom diameter) at both top and bottom by means of rubber tubing. The paper strips are hung from the top by means of a stainless-steel ring and wire hooks. The lower Buchner funnel is connected to a water pump.

G. RUSSELL

5310. **Direct quantitation on paper chromatograms of radioactivity from fatty acids labelled with carbon-14.** H. J. M. Hansen (Danish Atomic Energy Comm., Res. Establishment, Risö, Denmark). *Acta Chem. Scand.*, 1961, **15** (3), 670–674 (in English).—A statistical analysis of results obtained from experiments with reversed-phase chromatography shows that no bias in scanning is caused by variations in spot size or the type of fatty acid. A bias was found to be caused by variations in the distribution of the spot in a direction perpendicular to the plane of the paper. It is therefore recommended to scan low-energy labelled paper chromatograms from both sides of the paper and average the results. J. HALL

5311. **Improvements relating to gas chromatography.** Perkin-Elmer Corp. Brit. Pat. 872,259, date appl. 7.10.59. U.S.A., date appl. 8.8.58.—A gas-chromatographic analyser comprises two contiguous compartments, one of which carries a valve, for controlling the flow of the sample and carrier gases, and supports a chromatographic column on a wall having good thermal conductivity. The second compartment contains a motor connected with the valve so as to change it from a position in which the carrier gas is directed through the column and the sample gas to a metering device, to a second position in which a measured quantity of sample gas is directed from the metering device through the column and thence with the carrier gas to a thermal detector.

J. M. JACOBS

5312. **1,2,3-Tris-(2-cyanoethoxy)propane, a stationary liquid for gas-chromatography columns.** H. M. McNair and T. De Vries (Dept. of Chem., Purdue Univ., Lafayette, Ind., U.S.A.). *Anal. Chem.*, 1961, **33** (6), 806.—The solvent described, which is readily prepared from glycerol and vinyl cyanide (Brunson and Riener, *J. Amer. Chem. Soc.*, 1943, **65**, 23), shows great selectivity for olefins, aromatics, naphthenes, alcohols, esters, ethers and alkyl halides. It has a small affinity for paraffins. Retention vol. are given for 20 compounds at 153°, 100° and 55°, obtained on a 4-ft. column of the solvent supported on acid-washed Chromosorb, with a thermal-conductivity detector.

C. B. BAINES

5313. **Gas-liquid chromatography in qualitative analysis. I. An interpolation method for the prediction of retention data.** M. B. Evans and J. F. Smith (Natural Rubber Producers Res. Assoc., Welwyn Garden City, England). *J. Chromatography*, 1961, **5** (4), 300–307 (in English).—The glass columns used were 5 ft. long by 4 mm bore. The carrier gas was a mixture of H and N (3:1) and a flame-ionisation detector was used. The stationary phase was either Silicone Fluid 704, squalane or polyoxyethylene glycol 400 on acid-washed Celite or C22 brick (60 to 72 mesh); 10-μg samples were used and the column efficiency was 400 to 500 theoretical plates per ft. Compounds of the type R–X–R, R–X–R′ and R′–X–R′ were studied. If the respective retention times for these are $R_{1\cdot1}$, $R_{1\cdot2}$ and $R_{2\cdot2}$, it was found that, for many compounds, $\log_{10} R_{1\cdot2} = 0{\cdot}5\ (\log_{10} R_{1\cdot1} + \log_{10} R_{2\cdot2})$, provided that certain specified conditions are met. Experimental and calculated results are compared for aliphatic and aromatic hydrocarbons, olefins, monosulphides, disulphides, thiols, ketones, selenides, aromatic ethers, secondary alcohols and halides. G. RUSSELL

5314. **Investigation of the linearity of a stream splitter for capillary gas chromatography.** L. S. Ettre and W. Averill (The Perkin-Elmer Corp., Norwalk, Conn., U.S.A.). *Anal. Chem.*, 1961, **33** (6), 680–684.—An apparatus is described, and evidence, based on results obtained from hydrocarbon mixtures, is presented that demonstrates its effectiveness as a linear stream splitter over a wide range. The criteria of the linearity of the device are discussed. C. B. BAINES

5315. **A high-field emission ionisation detector for gas chromatography.** O. Wahlroos (Biochem. Inst., Helsinki, Finland). *Acta Chem. Scand.*, 1961, **15** (3), 708–709 (in English).—Modifications made to the detector tube of Haahti *et al.* (*Anal. Abstr.*, 1960, **7**, 5497) enabled it to be used at 18°. With an aluminium cathode, an increase of inter-electrode distance by ≃100% and a decrease of the cathode surface area to ≃0·05 of the stated dimensions led to a response roughly dependent on the organic vapour concn. in the argon. There was a time lag of several seconds and a "dead period" varying from 30 sec. to several minutes after a sample had passed through the tube. Higher fields were needed with tungsten or platinum cathodes, but no "dead periods" were observed. With all types of cathode, large oscillations of the signal current were frequently obtained until it was stabilised by inserting a fine metal gauze round the anode at the gas inlet. A diagram of the tube and a chromatogram obtained with it are given. Responses, of considerably differing intensities, were obtained with hydrocarbons, alcohols, aldehydes, ethers and esters. J. Hall

5316. **Analysis of gas-liquid chromatograms by a punched-card technique.** R. K. Tandy, F. T. Lindgren, W. H. Martin and R. D. Wills (Univ. of California, Berkeley, U.S.A.). *Anal. Chem.*, 1961, **33** (6), 665–669.—The technique described involves the analysis of fatty-acid methyl esters in which a β-particle ionisation detector is used. The results quoted indicate that the method is potentially applicable to all gas-chromatographic work. The procedure eliminates manual calculations, enhances accuracy and simplifies the manipulation and storage of data. C. B. Baines

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See also Abstract—**4918**, Papers from I.U.P.A.C. Congress, Lisbon, 1956.

Optical

5317. **Stepping mechanism for X-ray and neutron diffractometers and spectrometers.** M. H. Mueller, L. Heaton and E. W. Johanson (Argonne Nat. Lab., Argonne, Ill., U.S.A.). *Rev. Sci. Instrum.*, 1961, **32** (4), 456.—A stepping mechanism capable of very high positioning accuracy has been designed using as a basis a Slo-Syn motor. This is a permanent magnet-type a.c. motor with two field windings but, when d.c. is applied to the windings, the motor will lock into magnetic hold position and if the voltage is switched will advance 1/100, 1/200 or 1/400 of a revolution depending upon the switching used. The amount of rotation is controlled by a transistorised electronic stepping unit (details not given). G. Skirrow

5318. **Digital recording in spectrum analysis.** F. S. Brackett (Nat. Inst. of Arthritis and Metabolic Diseases, Bethesda 14, Md., U.S.A.). *J. Opt. Soc. Amer.*, 1960, **50** (12), 1193–1200.—Digital recording of spectra on magnetic tape in computer format is described and illustrated. N. E.

5319. **Spectroscopic apparatus.** Baird-Atomic, Inc. [Inventors: J. L. Saunderson and E. Dubois]. Brit. Pat. 869,257, date appl. 30.10.57.—In a direct-reading spectrometer the spatial relationships between the entrance slit, the grating and the exit slits are so critical that minor changes in, *e.g.*, ambient temp., may cause their misalignment. This misalignment is automatically corrected with precision and dependability by a servo-system, photo-electrically controlled by "monitor radiation" from a source other than the excited sample. J. M. Jacobs

5320. **Improvements relating to spectrophotometers.** Perkin-Elmer Corp. Brit. Pat. 869,194, date appl. 2.3.58. U.S.A., date appl. 4.3.58.—A system designed to define the optical paths within a radiant-energy spectrophotometer is described. Two beams of the radiant energy are directed from the source along paths which include reflecting optical elements. The beams traverse a space for the insertion of a sample before forming two identical co-planar images of the source, after which alternate portions of the beams are combined into a composite beam. J. M. Jacobs

5321. **The sensitivity of emission spectral analysis.** S. L. Mandel'stam and V. V. Nedler. *Optics and Spectroscopy*, 1961, **10** (3), 196–200; English translation of *Optika i Spektroskopiya*, 1961, **10** (3), 390–397.—The sensitivity limit, the smallest content of an element in a sample that can be determined, is defined as that content which gives a spectral-line signal several times as great as the random fluctuations of the background signal caused by the radiation detector. On this basis the arc and the hollow cathode as excitation sources are discussed. Theoretical estimates of these sensitivity limits are two or three orders of magnitude lower than those obtainable at present in the usual analytical procedures. It is suggested that the main potentialities in improving the sensitivity of spectroscopic analysis lie in devising more favourable conditions for transfer of atoms from the sample into the luminous cloud of the source, in improving the retention of atoms in the region of the source, and in improving methods for enriching the samples. B. S. Cooper

5322. **Effect of take-off angle on electron-probe calibration.** L. S. Birks and R. E. Seebold (U.S. Naval Res. Lab., Washington 25, D.C., U.S.A.). *Anal. Chem.*, 1961, **33** (6), 687–689.—Calibration curves are prepared for the nickel-iron and nickel-chromium systems at take-off angles of 6°, 20° and 90° using a calculation technique and 100% standards. Results show that the criterion of sensitivity to take-off angle is not the absolute value of the matrix absorption coeff., but rather the relative value compared with the self-absorption coeff. of an element for its own radiation. C. B. Baines

5323. **Simple glass variable-thickness cell for visible, ultra-violet and near infra-red spectrophotometry.** E. J. Breda and E. V. Kotkas (E. I. du Pont de Nemours & Co., Inc., Eastern Lab., Gibbstown, N.J., U.S.A.). *Anal. Chem.*, 1961, **33** (6), 816.—An inexpensive and versatile cell for solution thicknesses up to 5 cm is constructed from a hypodermic syringe, the neck of a graduated flask and silica windows. The cell functions satisfactorily with solvents such as aq. acid, alkali and various salt solutions and with alcoholic, ethereal and hydrocarbon systems. Aqueous ammoniacal solutions and 1,2-dichloroethane tend to dissolve the cement used in the construction. C. B. Baines

5324. **The use of pressed discs of potassium chloride in ultra-violet spectrophotometry.** V. A. Shlyapochnikov and V. I. Slovetskiĭ. *Optics and Spectroscopy*, 1961, **10** (2), 132; English translation of *Optika i Spektroskopiya*, 1961, **10** (2), 265.—The pressed-disc technique, widely used in infra-red spectroscopy, has been found to be of value in the ultra-violet region for samples for which suitable solvents are not available. Of several salts examined, KCl was the most suitable. The transmission of a disc $\simeq 1$ mm thick is $>60\%$ throughout the range 220 mμ to 320 mμ.

B. S. COOPER

5325. **Automatic sample changer for routine infra-red analysis.** J. W. Cassels, E. G. Brame and C. E. Day (Polychemicals Dept., E. I. du Pont de Nemours & Co., Inc., Wilmington 98, Del., U.S.A.). *Anal. Chem.*, 1961, **33** (6), 813.—An automatic 35-mm slide changer is adapted for use with a Perkin - Elmer spectrophotometer Model 21; 70 routine scans are obtained during a single unattended operation, equivalent to 24 hr. of operation for a 20-min. scan cycle.

C. B. BAINES

5326. **Preparation of potassium bromide discs for infra-red analysis.** J. Morris and S. J. van der Walt (Res. and Process Div. Dept., ISCOR, P.O. Box 450, Pretoria). *J. S. Afr. Chem. Inst.*, 1961, **14** (1), 16–21.—The results obtained from experiments based upon a short review of published experimental data are reported. It is concluded that no advantage is obtained by using extreme pressures (>7000 kg per sq. cm), whereas the drying of the halide under vacuum and evacuation of the die to about 2 torr before pressing minimised the intensity of absorption of adsorbed –OH groups.

E. G. CUMMINS

5327. **Analysis of the reproducibility of infra-red band intensities.** H. A. Szymanski and D. W. Teloh (Canisius Coll., Buffalo, N.Y., U.S.A.). *Anal. Chem.*, 1961, **33** (6), 814–815.—A study has been made of the reproducibility of intensities, and suggestions are given for the conversion of band intensities measured in one instrument into similar values for another instrument. Four different instruments are considered and an instrument constant is derived.

C. B. BAINES

5328. **Low-cost ordinate-scale expansion unit for the Perkin - Elmer infra-red spectrophotometer Model 21.** D. F. Westneat (Polychemicals Dept., E. I. du Pont de Nemours & Co., Inc., Wilmington, 98, Del., U.S.A.). *Anal. Chem.*, 1961, **33** (6), 812–813.—An inexpensive and easily constructed accessory is described, which can produce up to a 50-fold expansion in the recorded intensity of absorption bands. A scale-expansion circuit is given and the mounting of a potentiometer on the spectrophotometer is described.

C. B. BAINES

5329. **Use of infra-red band contours in applied spectroscopy.** H. A. Szymanski (Canisius Coll., Buffalo, N.Y., U.S.A.). *Anal. Chem.*, 1961, **33** (6), 815–816.—A study is made of the contours observed for a large number of olefins which show a distinct band between 10 and 15 μ. Sodium chloride optics are used and the bands are divided into three classes. The positions of the bands are influenced by the number of substituents at the ethylenic linkage.

C. B. BAINES

5330. **Filter photometry using cadmium sulphide detectors.** G. A. Rost (Quality Control and Analytical Lab., The Dow Chemical Co., Rocky Flats Plant, Denver, Colo., U.S.A.). *Anal. Chem.*, 1961, **33** (6), 736–738.—Designs and diagrams are given for an inexpensive, small and rugged filter photometer with a cadmium sulphide detector. The advantages and disadvantages of the detector are discussed. Photometers in which the separation of the sensing unit and the read-out meter is possible are of primary interest for achieving maximum utility for glove-box operation and remote monitoring. Similar instruments have been used for the analysis of soln. (0·03 to 5%) of Pu^{III} and Pu^{IV}.

C. B. BAINES

5331. **Investigations with a Zeiss flame photometer with a spray chamber.** E. Pungor, B. Weszprémy and M. Pályi (Inst. for Inorg. and Org. Chem., L. Eötvös Univ., Budapest). *Mikrochim. Acta*, 1961, (3), 436–443 (in German).—Two atomisers in parallel fed mist into an acetylene - air flame, via either a Y-tube or the chamber supplied with the Zeiss instrument. One atomiser was supplied with aq. 2mM-NaCl, the other with 2mM-KCl containing up to 91% of methanol, ethanol or propanol. It was found that the amount of mist that reached the flame increased with increasing alcohol content. Increasing the alcohol content resulted in a very greatly increased K emission, and a slightly decreased Na emission. The results were in good agreement with a second-order rate of precipitation of the mist. The rate constant increased with the chain length of the alcohol. An empirical expression for the rate of flow of soln. through the atomiser as a function of alcohol concn. was obtained.

R. M. ROWLEY

5332. **Improvements in and relating to polarimetry and polarimeters.** J. Bor. Brit. Pat. 868,359, date appl. 29.4.58.—The angular setting of a rotatable Nicol-prism arrangement in a polarimeter is determined by balancing the intensity of radiation transmitted through the polarimeter against the intensity of a beam of radiation not transmitted through the polarimeter. The accuracy of the determination of the angular setting for complete extinction of the transmitted radiation is thereby increased.

J. M. JACOBS

Electrical

5333. **Improvements in or relating to apparatus for indicating the metal oxide content of a liquid metal.** United Kingdom Atomic Energy Authority [Inventor: L. R. Blake]. Brit. Pat. 873,912, date appl. 3.6.58.—The presence of metal oxide, gas bubbles, etc., in a liquid metal flowing through pipework is indicated by measuring small changes in the electrical resistivity of the liquid metal.

J. M. JACOBS

5334. **Improvements in or relating to alternating-current polarographs.** United Kingdom Atomic Energy Authority [Inventor: G. C. Barker]. Brit. Pat. 870,451, date appl. 26.6.57.—An a.c. polarograph is described that comprises means for modulating the a.c. component of the current flowing in the polarograph cell, and means for measuring the currents resulting from the demodulation of the a.c. component caused by electrode reactions as a result of the non-linear variation of their rates with potential.

J. M. JACOBS

ABBREVIATIONS

Certain abbreviations in everyday use are not included in the following list. When any doubt might arise from the use of an abbreviation or symbol the word is printed in full.

alternating current	a.c.
ampere	amp.
Ångström unit	A
anhydrous	anhyd.
approximate, -ly	approx.
aqueous	aq.
atmospher-e, -ic	atm.
boiling-point	b.p.
British thermal unit	B.Th.U.
calorie (large)	kg-cal.
calorie (small)	g-cal.
centimetre	cm
coefficient	coeff.
Colour Index	C.I.
concentrated	conc.
concentration	concn.
constant	const.
crystalline / crystallised	cryst.
cubic	cu.
current density	c.d.
cycles per second	c/s
density	ρ
density, relative	d or wt. per ml
dilute	dil.
direct current	d.c.
distilled	dist.
electromotive force	e.m.f.
equivalent	equiv.
ethylenediaminetetra-acetic acid	EDTA
gram	g
gram-molecule	mole
half-wave potential	$E_{\frac{1}{2}}$
hour	hr.
hydrogen ion exponent	pH
infra-red	i.r.
insoluble	insol.
international unit	i.u.
kilogram	kg
kilovolt	kV
kilowatt	kW
liquid	liq.
maxim-um, -a	max.
melting-point	m.p.
microgram	μg (not γ)
microlitre	μl
micromole	μmole
micron	μ
milliampere	mA
milli-equivalent	milli-equiv.
milligram	mg
millilitre	ml
millimetre	mm
millimicrogram	mμg
millimolar	mM
millivolt	mV
minute (time)	min.
molar (concentration)	M
molecul-e, -ar	mol.
normal (concentration)	N
optical rotation	α_l
ounce	oz
parts per million	p.p.m.
per cent.	%
per cent. (vol. in vol.)	% (v/v)
per cent. (wt. in vol.)	% (w/v)
per cent. (wt. in wt.)	% (w/w)
potential difference	p.d.
precipitate (as a noun)	ppt.
precipitated	pptd.
precipitating	pptg.
precipitation	pptn.
preparation	prep.
qualitative, -ly	qual.
quantitative, -ly	quant.
recrystallised	recryst.
refractive index	n_λ^t
relative band speed	R_F
relative humidity	r.h.
revolutions per minute	r.p.m.
saponification value	sap. val.
saturated calomel electrode	S.C.E.
second (time)	sec.
soluble	sol.
solution	soln.
specific gravity	sp. gr.
specific rotation	$[\alpha]_\lambda^t$
square centimetre	sq. cm
standard temp. and pressure	s.t.p.
temperature	temp.
ultra-violet	u.v.
vapour density	v.d.
vapour pressure	v.p.
volt	V
volume	vol.
watt	W
wavelength	λ
weight	wt.

In addition, the following symbols may be used in conjunction with numerical values or in mathematical expressions—

greater than	$>$	less than	$<$
not greater than	$\ngtr$	not less than	$\nless$
is proportional to	$\propto$	of the order of, approximately	$\simeq$

The principal Pharmacopoeias are denoted by B.P., U.S.P. or D.A.B., together with the identifying roman numeral or year.

Valency states are represented by a superscript roman numeral, *e.g.*, Fe^{II}, Mo^{V}. Substances in the ionic state are represented by Na^+, Fe^{2+}, Fe^{3+}, etc., for cations and by Cl^-, SO_4^{2-}, PO_4^{3-}, etc., for anions.

The chemical nomenclature used in the abstracts is that recommended by The Chemical Society ("Handbook for Chemical Society Authors", The Chemical Society, London, 1960), and may differ from that used in the original articles.

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CONTENTS

	Abstract
General Analytical Chemistry	
Reviews; reagents; methods of general application	4918
Inorganic Analysis	
General methods; elements (in order of the Periodic Table); minerals; industrial products	4923
Organic Analysis	
Elements and radicals; organic compounds; industrial products	5035
Biochemistry	
BIOLOGICAL FLUIDS; ANIMAL AND VEGETABLE TISSUES	5110
PHARMACEUTICAL ANALYSIS	5231
FOOD	
Food additives; beverages; edible oils and fats; vitamins	5247
SANITATION	
Air; water; sewage; industrial wastes	5279
AGRICULTURAL ANALYSIS	
Soil; fertilisers; herbicides; pesticides; animal feeding-stuffs	5286
General Technique and Apparatus	
GENERAL	5295
CHROMATOGRAPHY; ION EXCHANGE; ELECTROPHORESIS	5305
OPTICAL	5317
ELECTRICAL	5333

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